FINAL REPORT

on

EVALUATION OF SOIL CONTAMINANTS AND CONCENTRATIONS IN CROP PRODUCTION FIELDS

to

WHIRLPOOL CORPORATION - CLYDE DIVISION

January, 1990

BATTELLE
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Columbus, Ohio 43201-2693

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INTRODUCTION

Environmental engineers at Whirlpool's Clyde, Ohio division have been attempting to identify the reason or reasons for partial crop failure in a corn field that borders their Amert site. In light of this, Whirlpool requested that Battelle visit the site and collect soil and plant samples for analysis. During the late summer of 1988, Battelle collected and analyzed soil samples. At that time the field was planted in corn but the corn was fully mature and plant analysis would have been useless. During 1989, both corn and plant samples were collected for analysis. While most of the following text deals with the work done in 1989, some overlap with the 1988 work is included since nearly similar results were obtained from both studies.

MATERIALS AND METHODS

On August 10, 1989, soil and corn-leaf samples were collected from a corn field located on the Amert Schrader farm (Figure 1, Source: Soil Survey of Sandusky County, Ohio. 1983, Sheet No. 52). The corn field borders Whirlpool's Amert site to the North. A sketch of the corn field identifying the sampling locations is shown in Figure 2. Soil samples were collected from the same field that was sampled as part of a previous study conducted for Whirlpool (Zwick and Means, 1989).

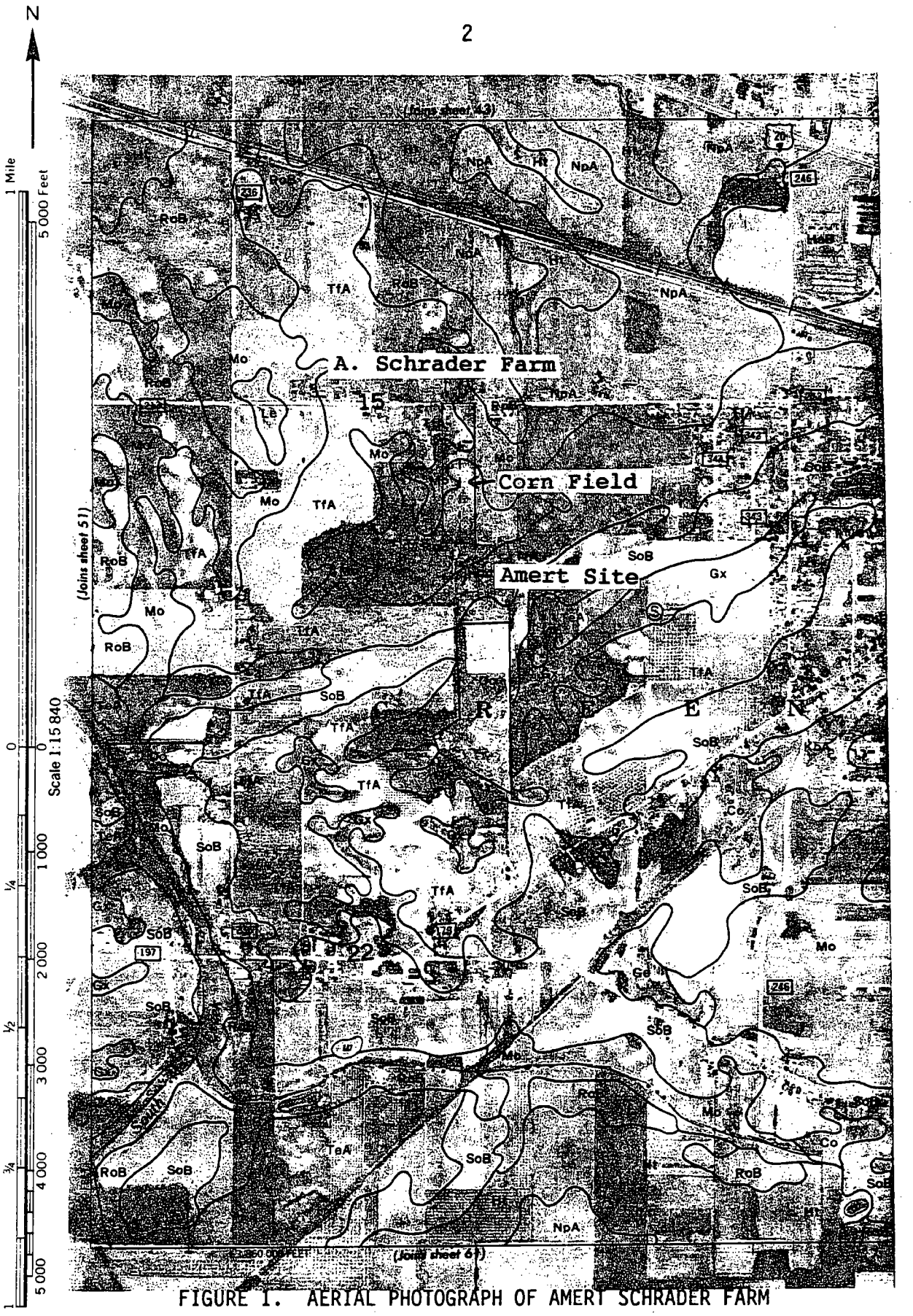
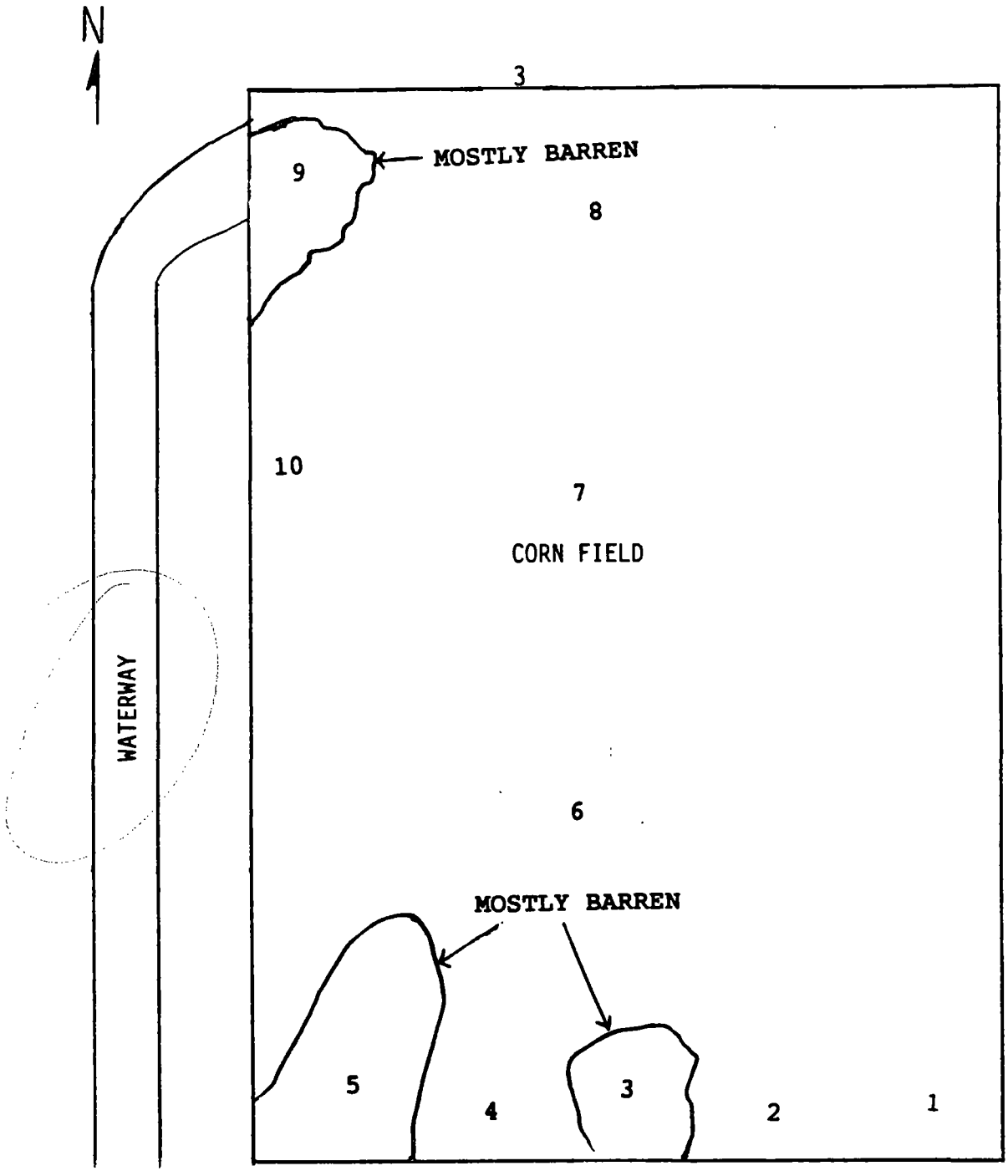


FIGURE 1. AERIAL PHOTOGRAPH OF AMERT SCHRADER FARM



Corn Field not drawn to scale

X: AMERT SITE

FIGURE 2. SAMPLING LOCATIONS IN CORN FIELD

Generally, surface drainage from the Amert site enters the south end of the ≈5 acre corn field, and generally flows to the north. Therefore, sampling locations were selected on an east-west transect across the south end of the entire corn field and on a north-south transect along the length of the corn field. The corn field at the south end was approximately 200 feet wide and included five sampling locations (1, 2, 3, 4, and 5). Locations 3 and 5 were selected in areas of the corn field that were nearly barren. The plants that were in the nearly barren areas were at most two-feet tall, stunted, and yellowish in color. The nearly barren areas, locations 3 and 5, were each approximately 600 ft^2 in area. Locations 1, 2, and 4 all contained healthy corn plants, which was evident based on the deep-green color of the plants and their uniform height (≈7 feet) and uniform silking. Sampling locations 6, 7, 8, 9, and 10 were selected along the length of the corn field (≈1000 feet long), north of the south end sampling locations. Sampling locations 6, 7, 8, and 10 contained healthy corn while location 9 (≈900 ft^2) was mostly barren, however, a few healthy corn plants dotted this area. The healthy corn plants observed at location 9 would indicate that soil contamination levels varied in this location probably due to the nature of the water movement from the waterway identified in the previously cited study. Locations 3, 5, and 9 correspond to locations 6, 7, and 1 of the previously cited study. Locations 7 and 1 were also barren portions within the corn field during the previous study, while location 6 contained healthy corn during the previous study.

At each sampling location, with the exception of location 3, surface soil samples were collected to a depth of approximately 7 inches. At location three, three samples (0-12, 12-24, and 24-36 inches) were collected. The subsurface profile was sampled at location 3 to evaluate vertical movement of selected contaminants. Six cores were collected and composited into paper bags at each location except for the subsurface cores collected at site 3, which consisted of two composite cores for each depth. Twenty corn-ear-leaves were also collected at each of the locations, equally divided per location, and composited into two paper bags.

Soil samples were dried at approximately 40 °C and shipped to the Research Extension Analytical Laboratory (REAL), located in Wooster, Ohio where they were ground, homogenized, and analyzed. Analysis included pH; lime test index; available phosphorus, boron, and manganese; exchangeable

potassium, calcium, sodium, and magnesium; cation exchange capacity (CEC); percent base saturation of calcium, magnesium, and potassium; soluble salts; and total heavy metals. Total heavy metal analysis included copper, zinc, lead, nickel, chromium, and cadmium. The standard analytical procedures followed are provided in Appendix A.

One of the two composited leaf samples collected from each location was oven-dried at approximately 40 °C and analyzed by the REAL. Analysis of the dried and homogenized leaf samples included nitrogen, phosphorus, potassium, calcium, magnesium, manganese, iron, boron, aluminum, sodium, and total heavy metals (copper, zinc, lead, nickel, chromium, and cadmium). Refer to Appendix A for the analytical procedures followed.

The second set of composited leaf-samples was retained at Battelle for analysis. The leaf samples were stored at approximately 4 °C until analyzed. The leaves were cut into small pieces (≈1-inch sections) and the entire sample from each location was divided into six equal lots. Each lot was placed into a flask and deionized water was added at a rate of 20 mL/gram tissue. The mixture was heated at 75-80 °C for one hour and allowed to cool to room temperature. The mixture was mixed by hand and conductivity measurements were read on a 50-mL aliquot of the water extract.

RESULTS AND DISCUSSION

Table 1 includes soil analysis data for pH, boron, sodium, and total heavy metals. Appendix B contains copies of the complete soil and plant analyses reports.

Surface pH's ranged from 5.1 to 7.3. Relative to areas designated as healthy, higher pH values were observed in the nearly barren location 3, 5, and 9. No explanation for this is apparent. The average pH for the entire corn field was 5.9, and would indicate that lime should be applied to bring the soil pH within the range of 6.0 to 6.5, the pH range recommended for corn production in Ohio (Ohio Agronomy Guide, 1985).

Soil analysis data (Table 1) indicates that sampling locations 3, 5, and 9, identified as mostly barren, have elevated boron and sodium levels compared

TABLE 1. SOIL ANALYSIS DATA

Sampling Location	pH	Concentration[1], µg/g								Soluble Salts mmhos cm^{-1}
		Boron	Sodium	Lead	Cadmium	Nickel	Chromium	Copper	Zinc	
1	5.1	12	14	22	<0.33	16	21	6	73	0.71
2	5.7	18	48	18	<0.33	11	20	6	51	0.42
3(0-12")	6.8	43	144	19	<0.33	13	18	7	59	0.33
3(12-24")	7.7	31	98	<13	<0.33	12	14	3	41	0.20
3(24-36")	7.8	41	220	36	<0.33	51	17	25	105	0.16
4	6.0	7	24	23	<0.33	14	20	9	66	0.36
5	6.3	46	100	17	<0.33	17	24	6	53	0.72
6	5.5	2	10	22	<0.33	14	19	12	74	0.22
7	5.2	1	12	21	<0.33	14	18	7	64	0.34
8	5.6	1	11	23	<0.33	16	19	12	59	0.22
9	7.3	10	32	24	<0.33	18	18	20	64	0.34
10	5.8	3	22	29	<0.33	21	21	14	80	0.34

[1] Soil samples collected to approximately 7 inches (unless noted) in corn field at silking.

to other locations, which were designated as healthy. The only exception to this is location 2 which was identified as containing healthy corn but showed boron and sodium contents higher than the mostly barren sampling location 9. However, sampling location 2 contained less boron and sodium than the nearly barren sampling locations 3 and 5. The data also show higher boron levels in healthy areas on the south end (locations 1, 2, and 4) of the field compared with healthy areas sampled on the north-south transect (locations 6, 7, 8, and 10). This would be expected since the south end of the corn field borders Whirlpool's Amert site. The elevated boron levels at sampling location 9 would appear to be a result of boron-contaminated surface-water moving there via the water-way west of the corn field.

Subsurface boron and sodium levels at sampling location 3 were also elevated indicating that boron and sodium have leached into the subsurface layer. Typical plant-available boron and sodium levels for Ohio soils range from 0.5-1.0 and 9-23 μg/g, respectively, (Watson, M., REAL, telephone conversation). As shown in Table 1, these background levels were exceeded at most of the sampling locations.

Total heavy metal concentrations (Table 1) at all locations were typical of background levels for Ohio soils. Background levels for Ohio soils in concentration units of μg/g are: lead 19, cadmium 0.2, nickel 18, chromium 12, copper 19, and zinc 75 (Logan and Miller, 1983). Logan and Miller (1983) have indicated that levels that are 2 to 3 times background levels should be indicative of metal contamination. All soil samples had metal concentrations less than two-times background with the exception of soil collected from location 3 from 24-36 inches which contained lead at a concentration of 51 μg/g, which is 2.8 times background (Table 1).

Soluble salt levels, which ranged from 0.16 to 0.71 mmhos cm^{-1} (Table 1), did not correlate with the elevated levels of boron and sodium. The soluble salt levels were well below 3 mmhos cm^{-1}, the salinity level at which severe injury to existing plants would be expected (Watson, M., REAL, telephone conversation). These soluble salt levels are below 1 mmhos cm^{-1}, the level at which seed germination may be inhibited, and would indicate that salinity levels were not associated with the poor plant stand observed in the nearly barren locations of the corn field.

Plant analysis data (Table 2) for boron and sodium for sampling locations 3 and 5 closely follow that of the soil analysis data. As with the soil, the highest boron (1735 and 1668 μg/g) and sodium (40 and 33 μg/g) levels were observed in ear-leaves collected from barren spots 3 and 5, respectively. Leaf tissue concentrations of boron and sodium at locations 2 and 4, which were designated as healthy, were higher than boron and sodium tissue concentrations found at location 9, which was mostly barren, but contained a few healthy plants.

Generally, corn tissue samples from locations 3 and 5 also showed reduced uptake of nitrogen and phosphorus compared to locations designated as healthy. Tissue concentrations of nitrogen at locations 6 and 7 were also reduced, however, the corn appeared healthy in appearance and only slightly reduced yields would be anticipated from these locations. Nitrogen and phosphorus (P) concentrations below 2.76% and 3000 μg/g in corn ear-leaf tissue collected at silking is considered marginal if normal yields are to be expected (Ohio Agronomy Guide, 1985). Potassium concentrations would be considered sufficient for normal yields. Interestingly, potassium concentrations in leaf tissues collected from locations 3 and 5 were elevated compared to the areas designated as healthy (Table 2). No apparent reason for this increased uptake of potassium is evident.

As with the soil conductivity analysis data (Table 1), no clear elemental uptake patterns were evident from the conductivity analysis of the plant tissue extracts. In the literature, plant tissue conductivity analysis has been shown to be a good indication of the soluble salt uptake by plants. For example, the conductivity threshold for maize is 1.7 mmhos cm^{-1}, and per every unit beyond this threshold, there can be expected a 19.0% yield decrease for this crop (Maas and Hoffman, 1977).

The plant tissue conductivity values were above the threshold and with the exception of sampling location 1, were less than a unit above the threshold, warranting very little to no yield reduction due to the observed conductivity values. However, it is apparent that the stunted plants would not be capable of reaching physiological maturity and therefore would not be capable of producing normal yields. Seed germination tests conducted with corn during an earlier study indicated that soluble salts levels were not

TABLE 2. PLANT ANALYSIS DATA[1]

Sampling Location	Boron µg/g	Sodium µg/g	Nitrogen %	Phosphorus µg/g	Potassium µg/g	Soluble Salts mmhos cm^{-1}
1	95	16	2.80	2751	21,335	2.85
2	876	24	2.82	3187	29,038	2.43
3	1735	40	2.47	2113	32,870	2.67
4	285	17	2.80	2885	27,551	2.18
5	1668	33	2.58	2366	30,744	2.67
6	30	17	2.53	2843	20,835	1.93
7	24	19	2.48	2852	19,986	2.34
8	21	15	2.87	3222	17,909	2.26
9	166	12	3.12	3247	19,329	2.59
10	19	15	3.08	2999	15,403	2.04

[1] Corn ear-leaf samples collected at silking.

inhibitory to seed germination in soils that had salt levels similar to levels reported in Table 1 (Zwick and Means, 1989).

The reason that most of the corn plants were severely stunted in barren locations 3, 5 and 9 is most likely due to restricted root growth resulting from the elevated boron levels. Furthermore, the lack of plants in these locations most likely was a result of the boron killing the young seedling at the time of germination/emergence (Johnson, J., OSU Agronomy Department, personal communication). Corn, which is semi-tolerant to boron toxicity, has been reported to show boron-toxicity symptoms at a soil concentration of 5 μg/g (Mengel and Kirkby, 1979).

SUMMARY AND CONCLUSIONS

Most of the locations sampled in the corn field contained boron and sodium at concentrations that greatly exceeded typical levels reported for Ohio soils. The highest levels of boron and sodium were observed in mostly barren locations 3 and 5 at the south end of the corn field. Similar trends were observed in plant tissue collected from locations 3 and 5.

Soil pH was generally higher in the nearly barren locations. The metal concentrations were typical of background levels for Ohio soils. Salinity levels in both soil and plants were nearly at background levels.

While this study was not intended to define the full extent of the contamination, soil data obtained from this study and the 1988 study are similar, suggesting that a possible remediation program may need to be considered. One possible remediation program would involve measures which would stop water from moving from the Amert site to the corn field. Another possible solution to the problem would be to remove the boron contaminants from the Amert site.

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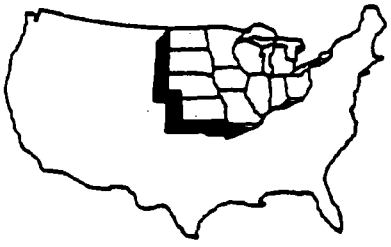
APPENDIX A

ANALYTICAL PROCEDURES

North Central Regional Publication No. 221 (Revised)

Recommended Chemical Soil Test Procedures

for the North Central Region



Agricultural Experiment Stations of
Illinois, Indiana, Iowa, Kansas, Michigan,
Minnesota, Missouri, Nebraska, North Dakota,
Ohio, South Dakota and Wisconsin,
and the U.S. Department of Agriculture
cooperating.

NORTH DAKOTA AGRICULTURAL EXPERIMENT STATION
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Bulletin No. 499 (Revised)
October 1988

FOREWORD

For nearly two decades, the NCR-13 Soil Testing Committee has provided the leadership in standardizing the procedures of soil testing laboratories in the north central region. They have conducted many sample exchanges and experiments to determine the influence of various laboratory procedures, such as extractant, sample size, soil: extractant ratios, and analytical instruments, on soil test results. As a result of these activities, the committee has arrived at the recommended procedures published herein.

The committee encourages all soil testing laboratories, public and private, within the north central region to use the procedures recommended in this publication. Experiments have shown that minor deviations in procedures may cause significant differences in test results. The adoption of these recommended procedures by all laboratories would be a major step towards improving the image and credibility of soil testing. If soil testing can be improved, the integrity and reliability of fertilizer recommendations based on soil tests would be improved markedly as well.

The NCR-13 committee wants it known that the publication of these tests and procedures in no way implies that research and innovation on methods of soil testing should stop. To the contrary, the committee strongly encourages increased research efforts to devise more accurate, more reliable and less costly soil tests. With the current pressure on farm profitability and the high cost of fertilizer, along with many soil related environmental concerns, it is more important than ever before that fertilizer be applied only where needed. The best hope of attaining this goal is better soil tests and better correlation with plant response. The NCR-13 committee stands ready to evaluate promising new tests and, as new procedures are proven to be an improvement, they will move quickly to revise their recommendations.

We wish to commend and thank the members of NCR-13 for their science, their dedication, and their spirit of cooperation in developing this bulletin. While all members of the committee are authors, and all have made significant contributions, Dr. William C. Dahnke should be singled out for special thanks as organizer and editor of the bulletin.

R. R. Davis
Administrative Advisor (1969-74)

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Administrative Advisor (1975-84)

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NCR-13

Introduction

William C. Dahnke*

For more than a century, soil and plant scientists have been developing methods for determining the levels of plant-available nutrients in soils. One of the first quick soil tests for "active" (available) nutrients was that of Daubeny (1) in 1845. It involved extracting the soil with carbonated water. His suggested test, however, was never put to practical use because of analytical difficulties. The first known fertilizer recommendations based on a soil test were made by Dr. Bernard Dyer (2) in 1894. He recommended that phosphate fertilizer be applied to soils releasing less than 0.01% P_2O_5(.0044% P) when extracted with 1% citric acid.

Since 1845 many extracting solutions have been suggested and tried. Some of the tests have proved to be very successful in spite of the fact that many different chemical forms of each nutrient occur in the soil, each having a different level of availability to plants.

Research efforts in developing soil testing as a useful guide to soil management have been extensive in soils and agronomy departments in the region. In most departments one or more prominent soils scholars have been associated with soil testing research over considerable periods of time. This, plus the fact that many soils in this region are amenable to corrective management, has resulted in the extensive use of soil testing in the NCR-13 region.

The preliminary work for this bulletin was done several years ago when a soil sample exchange was conducted among the member states. The results of this exchange indicated that differences in procedure were possibly causing significant differences in soil test results. A cooperative study among several of the states was conducted to determine the importance of procedural differences. For example, temperature, time and speed of shaking, and shape of extraction vessel were found to have an influence on the amount of phosphorus and potassium extracted (see Chapter 4). Soil scoops of the same volume but different depth and diameter were found to influence the amount of soil they hold. To solve this variability problem a standard soil scoop was suggested and is described in Chapter 2.

Another purpose of this bulletin is to describe the detailed procedures based partly on the above studies for soil pH, lime requirement, phosphorus, potassium, nitrate-nitrogen, calcium, magnesium, CEC, zinc, iron, manganese, copper, boron, chloride, sulfate-sulfur, soil organic matter, soluble salts and greenhouse media. We believe that use of these procedures by all public, private and industrial soil testing laboratories in our region will do much to reduce any confusion connected with soil testing and thus lend greater credibility to its role in the fertility management of soils.

The intent of the NCR-13 committee is to encourage continued work on procedures for these as well as other plant nutrients. As a new soil test or innovation is developed, it will be studied; and, if it offers improvements over a procedure in this bulletin, it will be adopted in place of or as an alternative to the one described herein. In addition to our research on soil testing procedures we plan to spend a substantial amount of time on soil test interpretation and fertilizer recommendations.

A word of caution to readers of this bulletin: A soil test is only as successful and usable for a region as the degree to which it is correlated and calibrated for the soils and crops of the area. The procedures described in this bulletin are especially suited to our region. Do not assume that they will work in your area without doing the necessary research.

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**North Dakota representative to NCR-13 committee and chairman of the publications subcommittee.*

in a refrigerated room (1 to 2°C) until the analyses are completed.

Sample Mixing and Measurement

1. Direct Method: This method is for use with coarse-textured soils or with soils containing small amounts of cementing materials such as clay and organic matter in an advanced state of decomposition, and also for undried soils where the slurry method cannot be used; i.e., in soils consisting mainly of undecomposed plant debris, and in peat soils which absorb their own weight of water or more.

Pass coarse-textured mineral soils through a 10-mesh screen and mix well. Determine the moisture percentage in the soil. Weigh out subsamples adding the actual moisture percentage to the desired weight of the subsample as expressed on an oven dry basis (100°) to give the weight of soil equivalent to the desired dry weight of soil for each test. With some balances, it may be more convenient to use one weight for all samples. Assume a moisture percentage in the range of 20-25%, whichever gives a rounded whole number. In the process of extraction of nutrients and determination of pH handle these subsamples as subsamples of dry soil. Adjust results for actual moisture percentages of the samples.

With undecomposed organic debris and spongy peat samples, measure out volumes equal to the volume occupied by the standard weight of a silt loam soil for each test. For P and K extraction or pH determination use the same procedures and reagents that are used with dry subsamples.

2. Slurry Method: Screening undried soil samples to achieve desired degree of fineness and mixing to obtain small representative subsamples takes time and may be nearly impossible in some soils. When extracting solutions are added to large clods of undried soils of high clay and organic matter content, the clods fail to disperse in the course of extraction and the results frequently are not reproducible. The following slurry method evolved from the attempts by J. J. Hanway and Kalju Eik to overcome the shortcomings of the direct method.

Pass the sample through a ¼-inch screen and mix it thoroughly. Estimate the moisture content of the moist soil samples and on a direct-reading balance weigh a subsample of the moist soil equivalent to 100 g of oven dry (110°C) soil into a mixing cylinder. Add enough distilled water to the soil in the cylinder to provide a total of 200 grams of water to 100 grams of dry soil. Stir the soil and water in the cylinders with electrical stirrers (Fig. 1) until all clods are broken up and a uniform suspension of soil in water is attained.

Place the cylinders on a rotator[1] (Fig. 2), and with calibrated automatic pipettes draw off subsamples of suspension in the amounts needed for each analysis while the cylinders are being rotated (Fig. 2). There are baffles attached to the inside wall of the cylinders to keep solid in suspension. Calibrate the pipettes for the desired amounts of dry soil equivalent with a number of soil samples by drying and weighing the solids actually delivered. The concentrations of phosphorus and potassium extracting solutions must be adjusted for the amount of water already in the subsamples. The precision of the slurry method, as measured by coefficient of variation, is comparable to the precision of dry methods.

[1]A rotator is a device consisting of a set of cups to hold the cylinders tightly (Fig. 2). These cups are rotated by a motor through a system of sprockets and a bicycle chain.

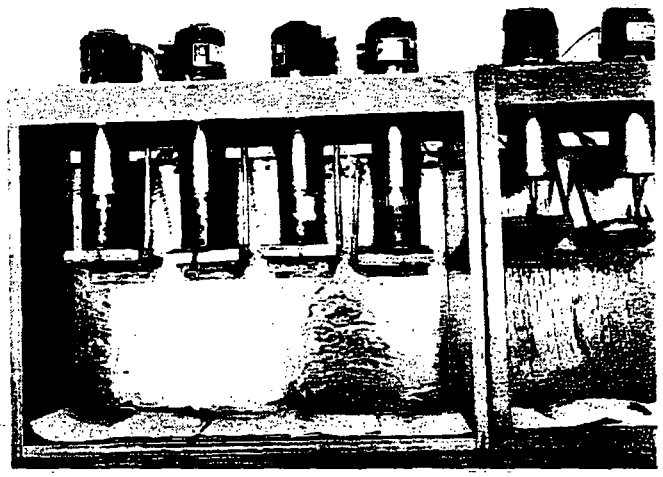

Figure 1. Electrical stirrers used in making a uniform soil suspension in the Slurry Method.

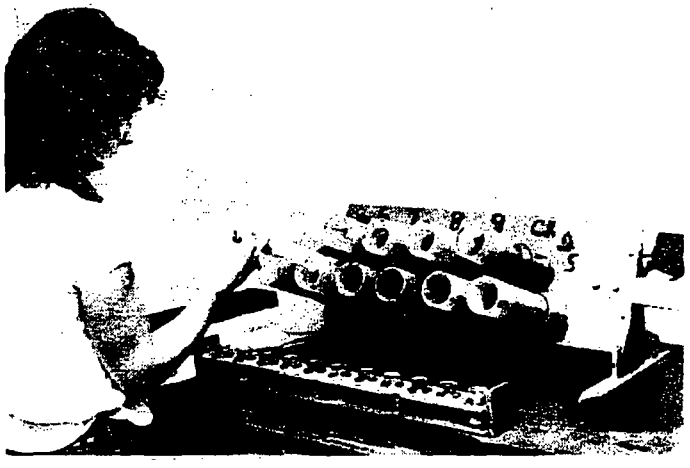

Figure 2. Drawing off a soil subsample for testing from cylinders on a rotator.

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scoops of two sizes were predominantly in use among the eleven states. These sizes were approximately 0.85 cc. and 1.0 cc. per gram of "typical soil." Scoop construction varied greatly, consisting of modified kitchen measuring spoons, calibrated copper tubing caps, and machined brass scoops. The basis for calibrating scoops was undocumented and vague. Conditions of wear and shape varied greatly, contributing further to the disarray in soil measurement. A search into the heritage of the two scoop sizes showed the 1.0 cc was introduced in the late 1950's in Illinois during a modernization[1] of the soil testing program and the 0.85 cc size was the scoop used by developers of early soil testing methods. The magnitude of the variations in test results was not as serious as variation in scoop size due to the small degree of dissociation of nutrient forms measured by available tests.

NCR-13 Scoop Development

The eleven NCR-13 members using volume measurement elected to adopt a standard size stainless steel scoop to minimize problems of variation among laboratories, wear of the scoop and contamination[2] of samples. Further, the committee members decided to continue using the weight-volume basis of reporting soil test levels since soil testing with these terms has appreciable farm acceptance in the North Central Region. The usual layman terms are pounds per acre, and conversion to the metric system can be pp2m or ppm designated with an asterisk to indicate scoop measurement.

Studies show the 0.85 cc size scoop test approximates a one-gram measure of "typical" soil. This is an empirical conclusion arrived at from the observation of several hundred volume-weight measurements on a wide range of soils. The "typical" soil is defined as a medial silt loam texture with 2.5% organic matter crushed to pass a 10-mesh screen. Bulk density of crushed "typical" soil approximates 1.18 compared with 1.32 for "undisturbed" soil. Experience with the 0.85 cc size scoop shows that soil test results on a soil sample measured with such a scoop when compared to analysis on a weighed sample differ by a factor equal to the difference in bulk density of the soil samples.

Table 1 shows the specifications for standard soil scoops as adopted by the NCR-13 regional soil testing and plant analysis committee. The scoops are illustrated in Figure 1.

PROCEDURE FOR USING SCOOP

Suggested procedure for using a soil scoop to measure soil is as follows:

1. Stir the crushed and screened sample with a spatula to loosen soil prior to measuring.
2. Dip into the center of the soil sample with the soil scoop, filling it heaping full without pushing against the side of the soil container.
3. Hold the scoop firmly. Tap the handle three times with a spatula from a distance of two to three inches.
4. Hold the spatula blade perpendicular to the top of the scoop and strike off excess soil.
5. Empty the scoop into an extraction vessel for the soil test.[3]

[1]*The scoop was machined of more durable material and hence was an improvement.*

[2]*Brass and copper scoops may contribute contamination in zinc and copper soil tests.*

[3]*Technique of measuring soil by scooping can be evaluated by weighing scoop contents. Precision not to exceed ± 10% should be expected.*

Table 1. NCR-13 Standard soil scoop specifications (Manufactured from stainless steel).

Scoop Size[1]	Scoop Capacity	Outside Diameter	Inside Diameter	Inside Depth
g	cc	in.	in.	in.
1	0.85	5/8	1/2	17/64
2	1.70	3/4	5/8	22/64
5	4.25	1	7/8	28/64
10	8.50	1-1/4	1-1/8	34/64

[1]Grams of soil in terms of the "typical" soil weighing 2,000,000 pounds per acre in the top 6⅔ inch layer.

NOTE: Commercially available from Soil Chem, Box 54, 114 S. Chicago, Rossville, IL 60963.

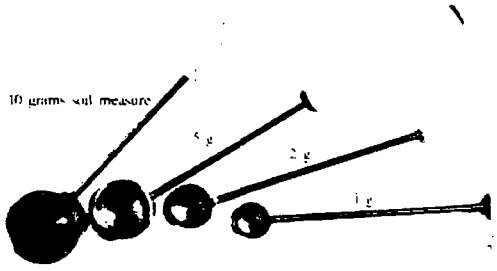

Figure 1. NCR-13 Standard Soil Scoops.

6. Calculate the analytical result using the scoop size (Table 1) as the assumed weight of soil and report soil test value in units of pounds per acre (acre will be understood to represent a volume measuring 43,560 square feet to a depth of 6⅔ inches).

Summary

Choice of a routine, rapid and accurate technique for measurement of the amount of soil for soil testing is an arbitrary one. In the experience of the NCR-13 committee, use of a scoop of the proposed size and shape will give soil test results comparable to weighed samples corrected for bulk density with a precision of ± 10%.

References

1. Bray, R. H. 1948. Requirements for successful soil tests. Soil Sci. 66(2): 83-89.
2. Melsted, S. W., and T. R. Peck, 1973. The Principles of Soil Testing. p. 13-21 *In* Walsh, L. M. and J. D. Beaton (ed.) Soil Testing and Plant Analysis. Rev. Ed. Soil Sci. Soc. of Amer., Madison, Wisconsin.
3. Mehlich, A. 1972. Uniformity of expressing soil test results: A case for calculating results on a volume basis. Comm. Soil Sci. and Plant Anal. 3(5): 417-424

Reference to commercial products does not constitute an endorsement, but is for the convenience of readers.

c. 955.8 g calcium chloride dihydrate

2. Add 9 liters distilled water, shaking vigorously during addition.
3. Weigh 36.0 g calcium acetate into a separate container and dissolve in 5 liters of distilled water.
4. Combine solutions 2 and 3, shaking during mixing and every 15 to 20 minutes for 2 to 3 hours.
5. Add 45 mL triethanolamine, shaking during addition and periodically thereafter until completely dissolved (may take up to 8 hours).
6. Dilute to 18 liters with distilled water, adjust to pH 7.50 using 15% NaOH, and filter.
7. Store in a container with the air inlet protected by drierite and ascarite to prevent contamination by water vapor and carbon dioxide. Avoid excessive agitation of the solution after pH adjustment.

Measuring soil-buffer pH

1. Add 10 mL of SMP buffer solution to the soil-water slurry saved from the pH determination.
2. Place in a mechanical shaker, close tightly, shake at 250 excursions/min for 10 minutes, and let stand for 30 minutes. (15 minutes shaking and 15 minutes standing is also acceptable, see Table 1).
3. Swirl and read the pH. Read to the nearest 0.01 pH unit, particularly if using the double-buffer option described below.
4. Use the resulting soil-buffer pH to determine the lime requirement from Table 2 or from local data.

Note: The lime requirement values given in Table 2 are based on average results of studies using a wide range of differing soils. They are intended as guidelines when no more specific information is available. Despite their usefulness in most situations, best results will be obtained when local calibration information is used.

Double-buffer Option for the SMP Test

A double-buffer option for the SMP test has been developed which may improve the accuracy of predicting lime needs on soils with low lime requirements (4,5). This procedure essentially develops the lime requirement - buffer pH relationship for each individual soil, rather than using the generalized relationship given in Table 2. It can be run as a simple add-on procedure to the standard SMP method. In contrast to the standard single-buffer method, certain properties of each batch of buffer should be determined precisely before using the double-buffer procedure. These properties may vary from one batch to the next, and their determination should be included in every preparation.

A. Preliminary Steps

1. Prepare an HCl solution of sufficient concentration that 1 mL will lower the pH of 10 mL of SMP buffer from pH 7.50 to pH 6.00. This solution should be approximately 0.21 *M*.
2. Titrate 10 mL of the pH 7.50 SMP buffer with HCl to determine the meq acidity neutralized per unit change in pH. Record this value as a_1. It should be approximately 0.137.
3. Add 1 mL HCl (prepared in step 1) to 10 mL of the pH 7.50 buffer, mix, let stand for 30 minutes, and titrate with HCl to determine meq acidity neutralized per unit change in pH. Record the value as a_2. This should be approximately 0.129.

B. Procedure for Double-buffer option

1. Record the buffer pH from the standard SMP test as pH_1.
2. Add 1 mL of the HCl solution (prepared in step A.I.) to the soil-buffer mix and repeat the 10 minute shaking and 30 minute standing (or 15 minute shaking and 15 minutes standing).
3. Read the pH of this mixture and record as pH_2.
4. Calculating the lime requirement
   a. Calculate the acidity to be netralized to achieve desired pH in meq/5g sample:

      $a_f = [(7.5\text{-}pH_1)a_1\text{-}(6\text{-}pH_2)a_2]$

      $[(pH_f\text{-}pH_2)/(pH_1\text{-}pH_2)] + (6\text{-}pH_2)a_2$

      a_f = acidity to be neutralized

      pH_f = desired pH

      All other values defined in Procedures A and B

   b. Calculate the lime requirement as follows:

1. LR (meq/100g) = $33.8a_f - 0.86$

Table 1. Effects of time of shaking and standing following shaking on average soil-buffer pH of 15 U.S. soils.

Shaking time	Standing time (minutes)			
	0	15	30	45
(min)	---------- pH ----------			
5	6.32	6.24	6.20	6.21
10	6.30	6.27	6.19	6.22
15	6.25	6.19	6.21	6.19
20	6.21	6.20	6.14	6.20

Table 2. Lime required to bring soils to desired pH as determined by SMP buffer pH.

SMP soil-buffer pH	Desired soil pH			
	Mineral soils			Organic soils
	7.0	6.5	6.0	5.2
	---------- tons pure $CaCO_3$ / acre* ----------			
6.8	1.1	0.9	0.8	0.6
6.7	1.8	1.6	1.3	1.0
6.6	2.4	2.0	1.7	1.3
6.5	3.1	2.6	2.1	1.7
6.4	4.0	3.4	2.8	2.1
6.3	4.7	4.0	3.3	2.5
6.2	5.4	4.6	3.7	2.9
6.1	6.0	5.0	4.1	3.2
6.0	6.8	5.7	4.7	3.6
5.9	7.7	6.5	5.3	4.1
5.8	8.3	7.0	5.7	4.4
5.7	9.0	7.6	6.2	4.7
5.6	9.7	8.2	6.7	5.2
5.5	10.4	8.8	7.2	5.5
5.4	11.3	9.6	7.8	6.0
5.3	11.9	10.0	8.2	6.3
5.2	12.7	10.7	8.7	6.7
5.1	13.5	11.4	9.2	7.1
5.0	14.2	12.0	9.7	7.5
4.9	15.0	12.7	10.2	7.8
4.8	15.6	13.1	10.7	8.2

* Per 2,400,000 lb. soil (8" furrow slice). Actual lime rate should be corrected based on characteristics of specific liming material to be used and depth of tillage.

I. The Effect of Speed on the Extractability of P and K in Erlenmeyer Flasks and Wheaton Bottles.

1. Extraction Procedure: 1-g samples were weighed in triplicate into 50-mL Erlenmeyer flasks and 30-mL Wheaton bottles. 10-mL of Bray P-1 or 1*M* ammonium acetate, pH 7.0 solution was added to both types of vessels and shaken on an Eberbach reciprocating shaker for 5 minutes at each of the three different speeds.
2. Bray P-1 Extractions
   a. Erlenmeyer flasks

      Equivalent amounts of P were extracted at 160 and 210 epm. By increasing the speed to 260 epm there were no statistical increases that were greater than 4.5%. The range in test values for all soils was 16.0 to 197 pounds per acre.
   b. Wheaton bottles

      Equivalent amounts of P were extracted at 160 and 210 epm. By increasing the speed to 260 epm there were no statistical increases greater than 4.7%. The range in test values for all twelve soils was 15.7 to 197 pounds per acre.
   c. Overall, the extraction of Bray P-1 between Erlenmeyer flasks and Wheaton bottles was similar at all shaking speeds. This is in sharp contrast to the three soils in the 1974 NCR-13 study.
3. Ammonium Acetate K Extractions
   a. Erlenmeyer flasks

      Three soils out of twelve showed statistical increases in K of greater than 5% in going from 160 to 210 epm (7, 9 and 12%). The 210 rate also resulted in the highest K values for eleven soils, although most were not statistically significant. There was no improvement by increasing the speed to 260, but rather, a very slight overall decrease. The range in test values for all soils was 97.3 to 578 pounds per acre.
   b. Wheaton bottles

      Equivalent amounts of K were extracted at 160, 210, and 260 epm. None of the soils showed statistical increases greater than 4.5% by going to the next faster rate. Although the magnitude of differences was not significant, the 210 rate resulted in the largest number of soils with the highest K. This is a sharp contrast to the behavior of three soils in the 1974 NCR-13 study, which showed large differences between the 160 and 200 epm rate. As with the Erlenmeyer flask, there was no improvement by increasing the speed to 260 but, rather, a slight but not significant, decrease in extractable K for ten of the soils. The range in test values for all soils was 98.0 to 577 pounds per acre.
   c. Overall, at 210 epm, the extraction of K in the Wheaton bottle was slightly less (3%), but not statistically different than the Erlenmeyer flask. Also, the K values were 6-9% lower for the Wheaton bottles on four soils out of the twelve. There was a slight, but not significant, decrease for both vessels at 260 epm. Very high shaking speeds can result in the soil solution movement not being able to adequately respond to the rapid change in direction of the shaker. This effect is related to the length of the shaker stroke, and to the size and shape of the vessel in relation to the amount of solution in the vessel.
4. Recommendations: The results of this study supported the previous listed recommendations, except that the effect of speed between 160 and 260 epm did not show the large differences found in the 1974 study. The differences found between the 1974 and the 1988 study are most likely due to the different soils used in each study. The 1980 guidelines(1), listed previously, should still be followed because of the difference in behavior of some soils.

II. The Effect of Lab Temperature on the Extractability of P and K

1. Extraction Procedure: 1-g samples were weighed in quadruplicate and K was extracted with 10 mL of 1 *M* ammonium acetate, pH 7.0, and P with Bray P-1 solution, in 50-mL Erlenmeyer flasks for 5 minutes at a speed of 200 epm. The extractions were carried out entirely in a climate controlled chamber at three different temperatures: 24, 29, and 35°C (75, 85 and 95°F). Soils and extracting solutions were equilibrated at the test temperature overnight for the extraction. Calibration curves, and molybdate blue colorimetric determinations for P were performed at the same temperature as each of the extractions.
2. Bray P-1
   a. Effect of temperature on phosphorus extraction: Changes in temperature had a significant effect on the extractability of Bray P-1 phosphorus. As the temperature was increased from 24 to 29°C, the increase in P ranged from 0 to 20%, with an average and median increase of 8.8 and 6.7%, respectively. Increases for eleven of the fifteen soils were statistically different at the 5% level of significance. As the temperature went from 24 to 35°C, the range in increase was 8 to 126%, with an average and median increase of 43 and 31%. The largest percent increases were for the lower concentrations, eg., 9 pounds per acre extracted at 24°C compared to 20 pounds per acre at 35°C. The increases were statistically significant for all fifteen soils. Changes at the lower concentrations are most serious since this is the area that has the greatest effect on fertilizer recommendations.
3. Ammonium Acetate K
   a. Effect of temperature on potassium extraction: Changes in temperature had a negligible effect on the extractability of potassium. The range in terms of percent increase in going from 24 to 29°C varied from no change to 3.2%, with an average increase of 1.2%. Only one sample was statistically significant. As the temperature went from 24 to 35°C the range in increase was significant for six of the fiteen samples and varied from 0 to 12%, with an average and median increase of 2.4 and 2.9% respectively. Only one sample out of the nineteen showed an increase greater than 6%.
4. Recommendations: From the findings in these temperature trials showing that serious errors can result with the extraction of Bray P-1 under different temperature regimes, it is strongly recommended that laboratory temperatures must be controlled (suggest 24 to 27°C) to assure the repeatability of results over time and to achieve comparative results between laboratories.

SURVEY ON EXTRACTION PROCEDURES

In the original publication on this subject, reference was made to a 1971 survey of twelve NCR-13 laboratories on extraction procedures. A similar survey was repeated for this publication. Following is a summary of both surveys comparing responses of 1971 and 1988. During this time the

NITRATE ELECTRODE METHOD

The function of the nitrate specific ion electrode as explained by Dahnke (2) is similar to a conventional pH electrode, but instead of developing a potential across a glass membrane, a potential develops across a thin layer of water-immiscible liquid or gel ion exchanger that is selective for NO_3^- ions. This layer of ion exchanger is held in place by a porous membrane.

The aqueous internal filling solution contains fixed levels of NO_3^- ion and chloride ion and provides a stable potential between the inside surface of the membrane and the internal Ag/AgCl reference element. The NO_3^- electrode responds only to the activity of the free unassociated ions, not to NO_3^- ions which are bound to complexing agents. If the activity of the NO_3^- ion is greater in the sample solution than in the internal filling solution, there is a net diffusion of NO_3^- ions into the electrode; or if the activity is less than in the sample solution, there is a net diffusion out of the electrode. The diffusion of NO_3^- ions into or out of the electrode will continue until a state of equilibrium is reached, at which time the electrical potential developed across the membrane prevents any further net diffusion of NO_3^- ions.

The lower limit of accurate detention of the NO_3^- electrode is about 1 to 2 ppm NO_3^--N in solution. This fact largely determines the smallest soil to solution ratios that can be used. Oien and Selmer-Olsen (11) studied ratios (g to mL) of 5:50, 10:50, 20:50, 30:50 and 50:50. They found that the ratio of 5:50 was too large to determine NO_3^- accurately in most soils because the NO_3^- contents are too low. They report that the ratio of 20:50 can be used to determine accurately as little as 2 ppm of NO_3^--N. When they used the ration of 50:50, the NO_3^- values decreased slightly when expressed as mg NO_3^--N per 100 g dry soil.

As ionic strength increases, the activity of the NO_3^- ion decreases. For this reason, numerous extractants have been developed to dampen this effect. The modified extracting solution of Millham et al. (10) is listed here (see below). If chloride and nitrite (NO_2^-) are not serious interferences, the silver sulfate and sulfamic acid can be eliminated from this extractant. Using present day module type electrodes, many workers have found that this extractant is an improvement over water. The electrode can be placed in a filtrate of the extract or directly in the soil-water slurry.

Equipment

1. Nitrate ion sensitive electrode.
2. A pH/ion meter or pH-millivolt meter.
3. NCR-13 10-g scoop.

Reagents

1. Extracting Solution:
   a. Distilled water or
   b. Ionic strength adjusting solution — 0.01*M* $Al_2(SO_4)_3$, 0.02*M* H_3BO_3, 0.01*M* Ag_2SO_4, and 0.02*M* NH_2HSO_3 (sulfamic acid): Dissolve 67 g of $Al_2(SO_4)_3 \cdot 18H_20$, 12 g of H_3BO_3, 20 g of Ag_2SO_4 and 19 g of NH_2HSO_3 in water and dilute to 10 liters.
2. Standard nitrate solutions. To a 1000 ml volumetric flask, add 0.7221 g of oven dry KNO_3; make up to volume with extracting solution. This gives a solution containing 100 ppm of NO_3^--N.

Procedure

1. Measure 20 g of soil with an NCR-13 10-g scoop into a 100-mL cylindrical container.
2. Add 50 mL of extracting solution.
3. Shake for 5 minutes on a reciprocal shaker.

Table 1. Working standard solutions for NO_3^--N test.

Volume of 100 ppm stock solution	Final Volume	Concentration of NO_3^--N in working standards
mL	mL	ppm
1	100	1
5	100	5
10	100	10
15	100	15
20	100	20

4. Read the potential while suspension is being stirred with magnetic stirrer.
5. Record the millivolt reading (if using a calibration curve technique) or read the NO_3^--N concentration directly from a pH/ion meter.

CADMIUM REDUCTION METHOD

Principle

This method of determining nitrate reduces NO_3^- to NO_2^- using copperized cadmium. Once reduced, the NO_2^- is usually determined using a modified Griess-Ilosvay method. This method is based on the principle that NO_2^- reacts with aromatic amines (diazotizing agents) in acidic solutions to give diazo salts. These salts couple with aromatic agents to form colored azo compounds or dyes. The color intensity is then determined with a spectrophotometer.

The range of detection for soil extracts using this method has been reported from 0.2 to 15 ppm NO_3^--N (5). This should be a sufficient range for most soils without additional dilutions. Precision values of 2.1 to 3.4% (coefficients of determination) have been reported using a manual method (3).

Other advantages of this method include the sensitivity of the Griess-Ilosvay procedure. This allows sufficient dilution to effectively eliminate any colored extract interference. The procedure is relatively rapid using automated instrumentation. From 40 to 100 samples per hour can be analyzed using automated procedures after samples are extracted. Instrumentation, however, is relatively expensive. Manual methods for cadmium reduction have been described (3, 6, 9). An estimate of 36 samples (previously extracted) analyzed per hour using four columns was made by Dorich and Nelson (3). The color of the azo compounds are very stable (1). Nitrite-N is determined simultaneously with NO_3^--N. However, NO_2^- can be determined separately by not passing one aliquot of the extract through the reducer column. The NO_3^--N is then determined by subtraction. Alternatively, NO_3^--N can be removed from the extract by addition of sulfamic acid.

Extraction can be accomplished with 2*M* KCl (3) or with water (6).

A procedure will not be specifically described here because of the lengthy methods involved. Each instrument will have its specific literature and method following the above principles. The reader is referred to Keeney and Nelson (9) or Huffman and Barbarick (6) for manual cadmium reduction methods.

References

1. Barnes, H., and A. R. Folkard. 1951. The determination of nitrates. Analyst (London) 76:599-603.
2. Dahnke, W. C. 1971. Use of the nitrate specific ion electrode in soil testing. Commun. in Soil Sci. and Pl. Anal. 2(2):73-84.

Considerable attention has been given to the details of soil-solution ratio, type of extraction vessel, speed and time of shaking, as well as to the chemistry involved in developing this procedure. By using this procedure it should be possible to obtain satisfactory agreement among laboratories.

Equipment

1. Standard NCR-13 1-g soil scoop
2. Spectrophotometer or colorimeter capable of absorbance of 882 nm (wavelength 610 to 660 for Fiske-Subbarrow alternate)
3. Rotating or reciprocating shaker capable of at least 200 excursions per minute (epm)

Reagents

1. Extracting solution (0.025*M* HCl in 0.03*M* NH_4F): Dissolve 11.11 g of reagent grade ammonium fluoride (NH_4F) in about 9 liters of distilled water. Add 250 mL of 1.00*M* HCl (previously standardized) and make to 10 liter volume with distilled water. Mix thoroughly. The pH of the resulting solution should be 2.6 ± .05. Store in polyethylene.
2. Stock standard phosphorus solution (50 ppm P): Dissolve 0.2197 g of reagent grade potassium dihydrogen phosphate (KH_2PO_4), that has been dried in a desiccator, in about 25 mL of distilled water. Dilute to a final volume of 100 mL with extracting solution. Under refrigeration, this standard should be stable for 6months to a year.
3. Working standard solutions: According to Table 1, pipette appropriate volumes of 50 ppm stock standard phosphorus solution into proper volumetric flasks.Bring each flask to volume with extracting solution.

Table 1. Working standard solutions for Bray P-1 Test.

Volume of 50 ppm stock solution	Final volume	Concentration of working standard	Equivalent concentration in soil	
			Ascorbic acid	Fiske-Subbarow*
mL	mL	ppm P	ppm P	ppm P
1	250	0.2	2.0	2.0
1	100	0.5	5.0	5.0
2	100	1.0	10.0	10.0
4	100	2.0	20.0	20.0
6	100	3.0	30.0	30.0
8	100	4.0	40.0	40.0
10	100	5.0	50.0	50.0
12	100	6.0	——	60.0

*Standards appropriate for the range of each method of color development are listed under the respective columns.

Ascorbic Acid Method

4. Acid molybate stock solution. Dissolve 60 g ammonium molybdate, $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, in 200 mL of distilled water. If necessary, heat to about 60°C until solution is clear and allow to cool. Dissolve 1.455 g of antimony potassium tartrate in the molybdate solution. Add slowly 700 mL of concentrated sulfuric acid. Cool and dilute to a final volume of 1000 mL. This solution may be blue, but will clear when diluted for use. Store in the dark under refrigeration.
5. Ascorbic acid stock solution. Dissolve 132 g of ascorbic acid in distilled water and dilute to a final volume of 1000 mL. Store in the dark under refrigeration.
6. Working solution. Prepare fresh each day by adding 25 mL of acid molybdate stock solution to about 800 mL of distilled water, mixing, adding 10 mL of ascorbic acid stock solution and making to a final volume of 1000 mL.

Fiske-Subbarow Method

4. Acid molybdate stock solution. (P-B solution). Dissolve 75.25 g of ammonium molybdate, $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, in 500 mL of distilled water heated to 60°C. Cool the solution and mix with 1,500 mL HCl (sp. gr. 1.19, 37.5%). Dilute the solution to 2000 mL with distilled H_2O in a volumetric flask and store in a glass stoppered brown bottle to which 100 g of boric acid (H_3BO_3) has been added.
5. Dry reducing agent: Aminonaphthol-sulfonic acid (P-C powder). Mix 5 g of 1-amino-2-napthol-4-sulfonic acid with 10 g of sodium sulfite (Na_2SO_3) and 292.5 g of sodium pyrosulfite ($Na_2S_2O_5$). Grind the mixture to a fine powder. If stored in a cool place in a sealed brown bottle, this reagent will keep for a year. Otherwise discard after 6 months.
6. Dilute reducing agent (P-C solution). Dissolve 16 g of dry reducing agent in 100 mL of distilled water heated to 60°C. Cool and store in brown bottle. Make fresh every three weeks.

Procedure

1. Using a NCR-13 1-gram scoop, scoop soil without pressing the soil against the side of the container. Firmly tap the handle of the scoop three times with an 8-inch spatula and level off the soil by passing the spatula across the scoop, holding the spatula at an angle of 90° (see Chapter 2).
2. Add measured volume of soil to a 50-mL Erlenmeyer flask, tapping the scoop on the funnel or flask to remove all of the soil from the scoop.
3. Add 10 mL of extracting solution to each flask and shake at 200 or more epm for 5 minutes with the room temperature at 24 to 27°C. (see Chapter 4).
4. Filter extracts through Whatman No. 2 filter paper or through a similar grade of paper. Refilter if extracts are not clear.

Ascorbic Acid Method

5. Transfer a 2 mL aliquot to a test tube (or remove quantitatively all but 2 mL from the filter tube if color is to be developed in the filter tube).
6. Add 8 mL of working solution so that thorough agitation and mixing occurs.
7. Allow 10 minutes for color development. Read per cent transmittance or optical density on a colorimeter or spectrophotometer set at 882 nm. Color is stable for about 2 hr.
8. Prepare a standard curve by aliquoting 2 mL of each of working standards, developing color and reading intensity in

Fiske-Subbarrow Method

5. Transfer a 5 mL aliquot to a test tube (or remove quantitatively all but 5 mL from the filter tube if color is to be developed in the filter tube).
6. Add 0.25 mL acid molybdate solution (P-B solution). Shake to mix with filtrate.
7. Add 0.25 mL dilute reducing agent (P-C solution). Allow color to develop 15 minutes before reading samples. Read per cent transmittance or optical density on a colorimeter or spectrophotometer set at 660 nm within 45 minutes after adding reducing agent.
8. Prepare a standard curve by pipetting a 5 mL ali-

7. Olsen, S. R., C. V. Cole, F. S. Watanabe, and L. A. Dean. 1954. Estimation of available phosphorus in soils by extraction with sodium bicarbonate. USDA Circular 939:1-19.
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7. Recommended Cation Tests and Measures of Cation Exchange Capacity

J. R. Brown and Darryl Warncke*

Potassium (K), calcium (Ca) and magnesium (Mg) availabilities in soil are generally estimated by measurement of the water soluble and exchangeable forms. The amounts of K, Ca, and Mg in the soil solution are quite small relative to the amounts in the exchangeable form. Hence, the quantities of these three cations extracted in most soil test procedures are simply referred to as exchangeable K, Ca and Mg. Available K levels in soils of the region are important for determining the appropriate rates of supplemental K to apply. Calcium in most North Central Region soils is rarely limiting as a plant nutrient. The measurement of exchangeable Ca may be used with the measurements of the other exchangeable cations to calculate an estimate of the cation exchange capacity of soils and/or to calculate the percentage of base saturation as an index for the need to neutralize excess soil acidity. Magnesium deficiences have occurred with sufficient frequency in the region to justify testing for Mg. A determination of available Mg will be helpful in deciding when to use dolomitic limestone.

The literature abounds with methodology used to measure exchangeable cations and cation exchange capacity. These methods were recently condensed (3, 5). The reader is referred to these references for details of the most accurate and precise procedures for determining plant available and exchangeable cations.

Soil testing or quick testing compromises some degree of accuracy for speed of determination. Therefore, standard or reference methods of soil testing have been developed to estimate nutrient availability. These estimates are then calibrated for recommendations based upon field trials with crop species of interest. A reference method, such as the one described herein for potassium, must be calibrated for the soil-crop-environment continuum for which it is to be used. These calibrations carried out in the various states and/or soil association areas provide the date for interpretation of the soil tests in terms of fertilizer needs. Thus, if different crop-environment combinations give different yield responses in different soil association areas different recommendations may result for the same soil test level. Potassium extractable with neutral 1*M* NH_4OAc has been well calibrated with crop responses and supplemental K needs for the varied soils of the North Central Region.

The cation exchange capacity (CEC) of soils is important in determining the supplemental K needs and the appropriate quantities of soil applied herbicides to use. The precise determination of CEC is time consuming. Soil testing labs in the region have determined that estimation of CEC by summation of exchangeable K, Ca and Mg and neutralizable acidity is acceptable for most soils. Gelderman (2) reports that CEC measures by summation may be inflated in calcareous soils by dissolution of $CaCO_3$ in the neutral 1*M* NH_4OAc. Therefore sodium acetate may be used as the replacing solution in the determination of CEC in calcareous soils.

ESTIMATE OF AVAILABLE POTASSIUM

The following procedure is slightly modified from the "NCR-13 Exchangeable Potassium Procedure" as written by Carson in earlier editions of this publication (1).

Equipment

1. Standard NCR-13 1 or 2-g scoop
2. Automatic or semi-automatic extracting solution dispenser (10 or 20 mL)
3. Extracting flasks (50-mL Erlenmeyer or conical flasks)
4. Funnels (or filter holding devices)
5. Receiving receptacle (20 to 30-mL beakers or test tubes) Note: Most high volume soil testing labs have racks of extracting flasks, funnels and receiving receptacles designed to handle multiple soil samples at one time
6. Rotating or reciprocating shaker capable of 200 excursions per minute (epm)
7. Atomic absorption/emission spectrometer (set in the emission mode for K)

*Missouri and Michigan representatives to NCR-13 Committee.

8. Recommended Sulfate-Sulfur Test

E. E. Schulte[1] and Kalju Eik[2]

Testing soil for plant available sulfur in the North Central region is done most extensively in the western and northwestern part of the region where there are fewer industrial centers. Minnesota, Nebraska and Wisconsin have reported areas where more or less consistent responses to sulfur fertilization have been observed, usually on sandy soils low in organic matter (3, 18, 10). Soil test summaries often identify considerable numbers of soil tests of surface samples in the "low" category (Eik, K., 1980. Unpublished summary of soil test results from Iowa State University Soil Testing Lab, 1974-79; Schulte, E. 1986. Unpublished soil test summary, Wisconsin, 1982-85), but small or no crop response from sulfur fertilization is observed in field experiments (29).

Increasing interest in sulfur soil tests is reflected by the fact that several states of the region and two adjacent Canadian provinces have made the test available to the public, partly because of demand and in part because of the need to compile information and to develop techniques and test correlations. Three other states have developed or adopted tests for research purposes only. Unfortunately, fertilizer dealers have jumped on the bandwagon, recommending "insurance" applications of sulfur equivalent to amounts removed by crops and neglecting contributions from the atmosphere and subsoil.

THE NATURE OF AVAILABLE SULFUR

Most of the sulfur in the surface soils (95 to 98% in Iowa soils) occurs in organic combination (24, 31). The mineralization of this organic sulfur can be an important source of plant available sulfur.

Plants take up most of their sulfur as sulfate ions. They can also absorb SO_2 directly from the atmosphere (7, 16, 21, 26). In well drained, arable soils almost all inorganic sulfur occurs as the sulfate ion in combination with cations either in solution or precipitated as salts in arid regions, or it may be absorbed by 1:1 clays and hydrous oxides of iron and aluminum. Adsorption increases as soil pH decreases below pH 6.5 (13). The inorganic sulfur determines the nutritional status of the crop (19), since both the soluble and adsorbed fractions are considered available. In soil testing usually the inorganic sulfur or the inorganic and a portion of the organic sulfur is measured. However, inorganic sulfur is usually present in the surface soil only in small quantities at any time. It is continually undergoing changes by mineralization and immobilization by microorganisms, leaching, and additions from the atmosphere in gaseous form or in precipitation (11, 17, 25).

There may be available sulfur also in the lower horizons of the soil profile and, as with testing for nitrate-nitrogen, this should be taken into consideration in any testing program. The amount of sulfate-sulfur added in irrigation water may be considerable. Thus, measurements of inorganic sulfate-sulfur in surface soil at any time does not necessarily reflect all the sources of readily or potentially available sulfur. However, since the movement, retention, and absorption by plants of available sulfur occur predominately as sulfate, and since sulfate-sulfur can be measured with relative ease, it is the fraction that is usually measured. Mineralization is slow and difficult to measure for prediction purposes. The atmospheric contribution fluctuates with the season and is also impractical to monitor except on a regional basis. Extensive reviews of sulfur reactions in soils as related to its availability and measurements have been given by Barrow (2), Ensminger (4), Ensminger and Freney (5), Reisenauer (19, 20), Widdowson (29), and Harward and Reisnauer (8). Water soluble sulfate is usually extracted with salt solutions such as $CaCl_2$, LiCl, or NaCl (29). Pure water tends to deflocculate soil and may dissolve some non-available organic sulfur (22). When an appreciable amount of sorbed sulfate is present, phosphate solutions, usually $Ca(H_2PO_4)_2$ or KH_2PO_4, are used. The phosphate ion has been chosen because of its greater strength of adsorption than that of sulfate, nitrate, or chloride ions (20). The $Ca(H_2PO_4)_2$ is more widely used because the presence of the Ca ion depresses the solution of organic matter and produces a clearer extract than is obtained with KH_2PO_4 (6). Sorbed sulfate is also extractable by $Ca(OH)_2$ (30). The use of phosphate solutions may give low results in soils containing gypsum (22).

EXPERIENCE WITH SULFUR TESTS IN THE NORTH CENTRAL REGION

Ten solutions containing 0 to 20 ppm S in water were analyzed by eight NCR-13 state laboratories in 1986. Six labs used turbidimetric analysis; two analyzed the samples by ion chromatography; one lab utilized inductively coupled plasma (ICP) emission spectroscopy; and one lab used an autoanalyzer. The average error by the four methods was 10.2, 9.4, 0.3, and 0.4% of the known values, respectively (Schulte et al. 1986. Unpublished NCR-13 sulfur subcommittee report). The greatest relative error occurred with low sulfate concentrations using the turbidimetric procedure. Analysis by ICP gave the lowest overall error on these water samples, but soil extracts are likely to contain some forms of sulfur other than sulfate which would be included in ICP analysis. An autoanalyzer permits more control over analytical conditions.

Sporadic reponses to S, mainly by alfalfa, have been recorded in parts of North and South Dakota, Nebraska, Minnesota, and Wisconsin. Elsewhere in North Central region, S response has been minimal and non-existent. Atmospheric S and S from manure and subsoils are contributing sources of this element which may not be measured in the soil test. Bundy (Univ. of Wisconsin, 1988. personal communication) measured a two-year average of 20 lb of S per acre per year in precipitation in non-responsive areas of Wisconsin and half that amount in reponsive areas. A study of profile sulfate-S in six Wisconsin soils at eight sites gave amounts ranging from 11 kg/ha to 90 cm in a loamy sand to 179 kg/ha to 150 cm in a silt loam soil (27). The average profile S to 90 cm was 72 kg/ha. Sulfate-S throughout the profile was highly correlated with organic matter in the surface 30 cm and negatively correlated with pH at each depth sampled. Considering these sources of sulfur, it would be better to offer the soil S test only on a two-foot (or deeper) sample as is done with the nitrate test or to base S recommendations on a plant analysis.

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Klett-Summerson, plot readings against concentrations on linear graph paper. Read the ppm SO_4^{2-}-S in the soil extracts from the standard curve and multiply by 2.5 to get ppm in the dry soil sample.

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6. Carry a blank through the entire procedure with each run.
7. Read samples on the AA, ICP or DCP spectrometer unit using appropriate standards and instrument settings.
8. Report as ppm Zn, Fe, Mn or Cu in the soil: ppm in soil = ppm in extract X 2.

0.1*M* HCl EXTRACTION FOR ZINC

This procedure is based on the assumption that all or a portion of the soil zinc which will become available for plant uptake during a growing season is acid soluble. The quantity of acid-soluble zinc extracted serves as an index of zinc availability (12). The method is primarily for determining acid extractable zinc in neutral and acid soils. It is not suitable for alkaline soils with excess calcium carbonate because of the neutralization of the acid in the extracting solution unless some adjustment in the interpretation of results is made for the excess lime. Nelson, Boawn, and Viets (8) used "titratable alkalinity" as a correction. They also recommended repeated extractions on highly calcareous soils until the pH of the suspension is below 2.0. On calcareous soils the DTPA test is recommended over the 0.1*M* HCl procedure.

The 0.1*M* HCl test has been used quite successfully throughout the North Central Region and is presently used in Illinois, Michigan, Nebraska, Ohio, and Wisconsin. The 0.1*M* HCl test was developed with little coordination of procedures among states. Thus procedural differences exist among laboratories. Sorensen, et al. (11) showed that soil properties, soil to solution ratio, and length of extraction all affected the amount of Zn extracted. Variations in the method used must be taken into account when comparing Zn extracted and the interpretation of the results. The method presented here is the procedure developed at the University of Missouri (1).

Equipment

1. Atomic absorption spectrophotometer
2. Reciprocating or rotary shaker capable of at least 180 epm
3. Standard NCR-13 5-g stainless steel scoop (.85 cc/g)
4. Burets, 50-mL Erlenmeyer flasks, and filter funnels for extraction
5. Soil pulverizer and 10-mesh stainless steel sieve checked for zinc contamination

Reagents

1. Zinc-free, demineralized water
2. Redistilled 6*M* HCl (reagent grade conc. HCl could be used if Zn-free)
3. Zinc stock standard 1,000 ppm Zn
4. Working Zn standards: Prepare working standards by diluting aliquots of the stock (1,000 ppm Zn) with the extracting solution to cover the normal range in the soil. Standards of 0, 0.1, 0.5, 1.0, and 2.0 ppm will cover the critical range.
5. Extracting solution: Add 300 mL of the redistilled 6*M* HCl to about 10 liters of the zinc-free demineralized water and mix. Bring to a final volume of 18 liters with demineralized water and mix thoroughly.

Procedure

1. Air dry soil samples and crush to pass a 10-mesh sieve. (See Chapter 1 on sample preparation.)
2. Using an NCR-13 5-g scoop, scoop soil without pressing the soil against the side of the container. Firmly tap the handle of the scoop three times with an 8-inch spatula and level off the soil by passing the spatula over the scoop, holding the spatula at an angle of 90° (See Chapter 2).
3. Add the measured volume of soil to a 50-mL Erlenmeyer flask, tapping the scoop on the transfer funnel or flask to remove all of the soil from the scoop.
4. Add 20 mL of the extracting solution to each flask, place on the shaker, and shake at 180 epm or more for 30 minutes.
5. Filter through washed Whatman No. 2 filter paper (or equivalent) into 30-mL polypropylene beakers.
6. Carry a blank through the entire procedure with each run.
7. Determine Zn in the extracts with the AA unit using appropriate instruments settings and Zn standards.
8. Report results as ppm Zn in the soil: ppm in soil = ppm in extract X 4.

.033*M* H_3PO_4 EXTRACTION FOR MANGANESE

Three states in the North Central Region, Michigan, Ohio, and Wisconsin, are testing for Mn. Ohio and Wisconsin are using .033*M* H_3PO_4 (3) as their extracting solution. Michigan is presently using 0.1*M* HCl using a 1 soil to 10 extractant ratio. The method presented here is the procedure used in the Ohio Agricultural Research and Development Center Research-Extension Analytical Laboratory, Ohio State University, Wooster.

Equipment

1. Atomic absorption spectrophotometer
2. Reciprocating or rotary shaker capable of at least 180 epm
3. Standard NCR-13 1-g stainless steep scoop (.85 cc/g)
4. Burets, 50-mL Erlenmeyer flasks, and filter funnels for extraction
5. Soil pulverizer and 10-mesh stainless steel sieve checked for manganese contamination

Reagents

1. Manganese-free, demineralized water
2. Concentrated H_3PO_4 (85.5%)
3. Manganese stock standard of 1,000 ppm Mn
4. Extraction solution: Dilute 2.25 mL of concentrated H_3PO_4 to a volume of 1.0 liter with the Mn-free demineralized water.
5. Manganese standards: From the 1,000 ppm Mn standard, prepare working standards of 0, 0.5, 1.0, 2.0, 4.0 ppm Mn in the extracting solution. Additional standards may be necessary for samples low in extractable Mn.

Procedure

1. Air dry soil samples and crush to pass a 10-mesh sieve (See Chapter 1 on sample preparation.)
2. Using an NCR-13 1-g scoop, scoop soil without pressing the soil against the side of the container. Firmly tap the handle of the scoop three times with an 8-inch spatula and level off the soil by passing the spatula over the scoop, holding the spatula at an angle of 90° (See Chapter 2).
3. Add the measured volume of soil to a 50-mL Erlenmeyer flask, tapping the scoop on the transfer funnel or flask to remove all of the soil from the scoop.
4. Add 10 mL of the extracting solution to each flask, place on the shaker, and shake for 10 minutes at 180 epm.

10. Recommended Soil Boron Test

Maurice E. Watson*

Boron (B) is an essential nutrient to living plants (16). It has been characterized as a micronutrient because of the small quantity required to support optimum plant growth. Boron concentrations usually range from 5 to 80 ug per gram of dry plant tissue across plant species. The interval between deficiency and toxicity is narrow for most plant species. Some of the plants most senstive to B deficiency are celery, cauliflower, cabbage, Brussels sprouts, alfalfa, red clover, white clover, apple trees and pear trees.

Plants obtain boron from soluble B forms present in the soil. According to Lindsay (8), H_3BO_3 is the predominant B species in soil solution. Only at pH above 9.2 is the $H_2BO_3^-$ species expected to become predominant in soils.

Ideally the test for soil B should measure the form of B that is most important to plants. A successful soil B test must, however, be able to measure the amount of B that is immediately available, as well as that potentially available to plant roots. The better correlation is between plant absorption of B and the measure of B in the soil, the more useful is the test. The B test must be sensitive enough to allow accurate measurements of concentrations (either high or low) which are important to the plant. In addition, the test must be free from major interferences caused by other chemical constituents in the soil extract.

Berger and Truog (2) divided soil B into 3 categories: Total B; acid-soluble B (H_2SO_4); and water-soluble B. They concluded that water-soluble B correlated best with the incidence of black spot in garden beets. Work by Berger and Truog (3) as well as Starck, Truog, and Attoe (17) showed that all the B added to a mineral soil could be recovered with a boiling hot water extraction. In 1966, Miljkovic, Matthews, and Miller (10) related the uptake of B by sunflowers from eight different soils to the concentration of soil B as determined by a hot water extraction. Next to water-soluble B, clay content had the most influence on B uptake. These two variables in a curvilinear regression accounted for 79% of the variability in uptake from cultivated surface soil samples.

Hot water soluble B can be affected by many soil factors. Clays and oxides of iron and aluminum can fix boron (5,15). Also, the soil organic matter content has been shown to be important, particularly for soils that are not highly cultivated (13,9). The absorption of hot water soluble B by lucerne (alfalfa) was shown to be greater from coarse texture soils than from fine texture soils (20). A survey by Ouellette and Lachance (12) revealed that when lucerne was the dominant plant species, B deficiency occurred more frequently on coarse texture soils than on fine texture soils. They concluded that about 0.8 lb B/acre was necessary for normal growth of lucerne on fine texture soils compared to 0.5 lb B/acre on coarse texture soils. Variations in soil moisture and cultivation may also affect the amount of hot water soluble B present. Work by Winsor (21) showed that the concentration of hot water soluble B increased as the soil moisture level increased. The increase occurred both in virgin and cultivated soils, but was much more in virgin soils. The soil texture in this research was fine sand.

Methods that have commonly been used in the past to measure B have been those using quinalizarin and curcumin dyes (2,11). Azomethine-H has been used to complex the B in plant tissue and soil extracts (1, 22, 23, 6, 14). Kowalenko and Luvkulich (7) used a modified curcumin procedure and an acetate buffer extraction (pH 4.8) to measure available soil B.

The curcumin method has generally replaced the quinalizarin method because concentrated sulfuric acid is not required for curcumin. Disadvantages of the curcumin method are that water must be evaporated from the sample, and a great deal of handling is thus required. An advantage of the curcumin method over the Azomethine-H method is that of greater sensitivity. Methods that use inductively coupled plasma spectrographs (ICP) have greatly simplified the measurement of B.

HOT WATER EXTRACTABLE BORON

Equipment

1. Standard NCR-13 10-g soil scoop
2. Fiber digestion beakers (600ml)
3. Fiber digestion apparatus
4. Centrifuge
5. Plastic centrifuge tubes
6. Plasticware and/or low boron glassware when available

Reagents

1. Extracting Solution — Dissolve 1 g of $CaCl_2 \cdot 2H_2O$ in deionized water. Make to 1 liter volume with deionized water. Use high quality deionized water. $CaCl_2$ added to promote flocculation of the soil.

Procedure

1. Using an NCR-13 10-g scoop, scoop 10 g soil into a 600-mL fiber digestion beaker, add 20 mL of extracting solution, attach beaker to condenser of fiber digestion apparatus and boil for 5 minutes. Allow to cool slightly before removal. However, solution should remain warm.
2. Remove from apparatus and immediately transfer the suspension to a plastic centrifuge tube. Centrifuge for 15 minutes at 2700 *g*. Decant an aliquot from the supernatant extract for analysis.

MEASUREMENT OF BORON CONCENTRATION IN EXTRACT

Curcumin Method

Equipment

1. Spectrophotometer or colorimeter capable of measuring absorbance at 545 nm wave length
2. Plastic beakers
3. Waterbath

Reagents

1. Stock Standard (1000 ppm B): Dissolve 5.716 g of H_3BO_3 in about 900 mL of deionized water and dilute to 1 liter. Store in polyethylene bottle.
2. Working Standard (5 ppm B): Dilute 5.0 mL of 1000 ppm boron solution to 1000 mL of deionized water.
3. Ethyl Alcohol: Use absolute ethyl alcohol to avoid moisture problems which hinder proper color development.
4. Curcumin-Oxalic Acid Solution: Dissolve 0.04 g of curcumin and 5.0 g of oxalic acid in 100 mL of absolute

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Reagents

1. Stock Standard (1000 ppm B): Dissolve 5.716 g of H_3BO_3 in about 900 mL of deionized water and dilute to 1 liter. Store in a polyethylene bottle.
2. Working Standard (5 ppm B): Dilute 5 mL of 1000 ppm boron stock solution to 1000 mL with deionized water.

Procedure

1. If necessary, filter the centrifuged extracts through Whatman No. 2 filter paper or similar grade paper into small plastic tubes. Aspirate the supernatant into the standardized ICP.
2. Prepare B standards by accurately measuring exactly 20 mL of deionzed water into each of 5 plastic beakers. Add to each beaker exactly the volume of 5 ppm B standard as indicated in Table 3.

Table 3. Boron working standards for ICP method

Beaker	Deionized Water	Volume of 5 ppm Boron Standard	Boron in Solution	Boron in Soil
	mL	mL	ppm	lb/acre
1	20	0	0	0
2	20	0.5	0.12	0.48
3	20	1.0	0.24	0.95
4	20	2.0	0.455	1.82
5	20	3.0	0.65	2.61

3. Use the prepared standards to standardize the ICP across the full range of boron standards.
4. Carry a blank (a beaker containing only deionzed water) through the entire procedure to estimate any boron contamination that may be present.
5. Use a 40-second preburn setting and a 10-second integration time. Three 10-second integrations should be done.

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POTENTIOMETRIC KNOWN ADDITION METHOD

Direct reading of soil extracts with the solid-state Cl^- electrode has not been reliable across diverse soils and may give high readings (11). The electrode has worked well when used as an endpoint indicator in titrations. A more convenient alternative to potentiometric titrations is the potentiometric known addition method outlined here. It is particularly well suited for situations where occasional analysis for Cl^- concentration is needed since no calibration is necessary.

The basic approach of the method was reported by Bruton (12) for Cl^- and fluoride determination of phosphorus and involves measuring the electrode potential before and after addition of a known quantity of Cl^- to a sample. The change in potential is then related to sample concentration by assuming a Nernst-type relationship and a theoretical electrode response of 59.1 mV per ten-fold change in concentration. This electrode response should be verified by measuring potential after successive additions of the standard.

Equipment

1. Standard NCR-13 10-g scoop
2. Shaker
3. Solid-state Cl^- electrode and double junction reference electrode
4. pH/ion meter or pH-millivolt meter
5. Magnetic stirrer

Reagents

1. Extracting solution (0.5 *M* K_2SO_4): Weigh 87.0 g of K_2SO_4 into a 2-L volmetric flask. Bring to volume with distilled water.
2. Chloride standard stock solution (1000 ppm Cl^-): Dissolve 0.2103 g reagent grade KCl in approximately 50 mL of extracting solution. Bring up to 100 ml volume with extracting solution.
3. Chloride standard working solution (50 ppm Cl^-): Dilute 5 mL of stock solution to 100 ml with extracting solution.

Procedure

1. Scoop 10 g of crushed soil into 50-mL Erlenmeyer flask. Do duplicate or triplicate analyses. Include a blank sample.
2. Add 30 mL of extracting solution.
3. Shake for 15 minutes at 180 or more epm. Samples can be either filtered (No. 42 Whatman or equivalent), centrifuged, or left to settle to produce clear solutions.
4. Pipette 20 mL of the solution into a 50-mL beaker.
5. Place beaker on a stirrer, add magnet, and mix.
6. Immerse Cl^- electrode into the beaker and record MV reading once the meter has stabilized.
7. Add 2 mL of 50 ppm Cl^- solution and record MV reading when meter has stabilized.
8. The difference between the first and second readings is ΔE.
9. Sample concentration can be determined by either of the following approaches.

   A. Obtain a Q value which corresponds to the ΔE value from a known addition table that is usually supplied with the electrode. Multiply the Q value by the concentration of the standard (50 ppm) and subtract the blank concentration to determine the sample concentration.

   or

   B. Calculate the concentration directly as follows:

$$C = \frac{(C_S)(V_S)}{V+V_s}\left(10^{-\Delta E/59.1} - \frac{V}{V+V_s}\right)^{-1}$$

where: C = concentration of sample
C_s = concentration of standard
V = mL of sample
V_s = mL of standard

In this procedure the equation simplifies to:

$$C = \frac{4.545}{10^{-\Delta E/59.1} - 0.909}$$

Subtract the blank concentration from C.

10. Convert extract concentration to ppm in soil by multiplying by the dilution factor of 3.0.

ION EXCHANGE CHROMATOGRAPHIC METHOD

Chemically suppressed ion chromatography was introduced by Smith, Stevens, and Bauman (15) in 1975. The main advantages of this method are high sensitivity, the ability to separate and quantify similar types of ions (i.e. F^-, Cl^-, and Br^-), multiple element analyses and increased freedom from sample matric effect. Mosko (13) demonstrated some problems encountered in the analyses of a range of aqueous samples. The method of extraction of Cl^- from the soil is the same method used for NO_3^- (14). This allows the potential of multi-element analyses.

Equipment

1. Balance (0.01g)
2. Reciprocating shaker capable of approximately 200 epm
3. Dispenser or buret capable of dispensing 25 mL
4. 50-mL Erlenmeyer flasks and filter-funnel tubes
5. Mechanical vacuum extractor (Centurion) and syringes
6. Ion chromatography system including appropriate inline filters, column(s) and detector
7. Strip chart recorder and/or micro-computer aided data aquisition

Reagents

1. Extracting solution: Calcium hydroxide (saturated solution). Add calcium oxide to water (3 g per liter of distilled water); shake thoroughly. Filtration of the solution is desirable, but not necessary.
2. Eluant for ion chromatograph: Weight 0.2544 g of sodium carbonate and 0.2520 g sodium bicarbonate into a liter volumetric flask and make to volume with double distilled or distilled deionized (DDI) water.
3. Regenerant for chemically suppressed ion chromatography system utilizing a micromembrane suppressor. Add 1.5 mL concentrated sulfuric acid to a 1 liter volumetric flask and make to volume with DDI water.
4. Chloride stock standard solution (1000 ppm Cl^-): Dissolve 0.1648 g reagent grade sodium chloride in approximately 50 mL extracting solution. Make to 100 mL volume with extracting solution.
5. Chloride standard intermediate solution (100 ppm Cl^-): Pipette 10 mL of stock solution into a 100-mL volmetric flask and bring to volume with extracting solution.

12. Recommended Soil Organic Matter Tests

E. E. Schulte[1]

The importance of soil organic matter in supplying nutrients, contributing to cation exchange capacity, improving soil structure, etc., is well recognized. In some states, the organic matter content of the soil is used to adjust N, S, herbicide, and/or lime recommendations. The importance of soil organic matter in herbicide recommendations has rekindled an interest in organic matter analysis. In the future, the organic matter content of soil may also find use in calculating loading rates for sewage sludge and other wastes.

Organic matter determinations are usually based on one of two fundamental methods:

1. Weight loss on removal of the organic matter from the mineral fraction by:
   a. Oxidation with H_20_2
   b. Ignition
   c. Ignition after decomposition of silicates with HF
2. Determination of some constituent which comprises a relatively constant percentage of the organic matter such as:
   a. Nitrogen
   b. Carbon

The weight loss determinations are subject to errors caused by volatilization of substances other than organic materials (H_20, structural OH, CO_2 from carbonates) and incomplete oxidation of carbonaceous materials. Also, these methods are usually very time-consuming.

Recent interest in weight loss methods has arisen out of a desire to eliminate the use of chromic acid (below). Ball (3) compared the weight loss of 117 upland, 22 lowland, and 11 organic soils of North Wales at 850 and 375°C with organic matter determined by a modification of the Walkley and Black (11) procedure. Results at both temperatures were highly correlated with organic matter by the Walkley and Black procedure, but the lower temperatures was deemed preferable. Goldin (4) compared loss of weight on ignition of 60 non-calcareous soils of northwestern Washington and British Columbia with organic carbon determined with a Leco carbon analyzer ($R^2 = 0.98$). Storer (8) automated the procedure with a computerized weighing system.

Mehlich (5) extracted "humic matter" with 0.2 *M* NaOH — 0.0032 *M* DTPA — 2% ethanol; this method is used in North Carolina. Attempts to use this procedure on Wisconsin soils have resulted in poor reproductibility in replicate samples. It is believed that mobilization of clay may be partly responsible.

Estimation of organic matter by determination of total nitrogen is not widely used because of the relatively wide variation of nitrogen content in organic materials from different sources. However, carbon determinations are used extensively for this estimation, the carbon being determined by:

a. Dry combustion and measurement of CO_2 evolved (after removal of carbonates).
b. Chromic acid oxidation and measurement of CO_2 evolved (after removal of carbonates).
c. Chromic acid oxidation for determination of easily oxidized material (external heat applied).
d. Chromic acid oxidation for determination of easily oxidized material (spontaneous heating).

The dry combustion method measures total carbon whereas the chromic acid methods determine only that which is easily oxidizable. (The carbon in graphite and coal is not oxidized by chromic acid). The methods involving measurement of CO_2 evolved require special apparatus and are not well adapted to rapid analysis of a large number of samples. Consequently, the methods which involve chromic acid oxidation for the determination of easily oxidizable material are most widely used. These methods (c and d) differ primarily in the source and amount of heat used to drive the reaction. Method (c) utilizes an external source of heat which permits heating to a higher temperature that can be achieved with method (d) which derives its heat from the heat of dilution of concentrated H_2SO_4. Consequently, the reaction in method (c) is much faster and oxidation of the organic matter is more complete, but conditions must be carefully controlled to achieve reproducible results.

A temperature of approximately 120°C is obtained in the heat-of-dilution reaction of concentrated H_2SO_4 (2). This is sufficient to oxidize the active forms of organic C but not the more inert forms. Walkley and Black (11) recovered 60 to 86% of the organic C in the soils they studied. As a result of this and other work, a recovery factor of 77% is commonly used to convert "easily oxidizable" organic C to total organic C. Later work (1), however, showed that the recovery factor varied from 59 to 94%. The use of external heat, such as employed in the Schollenberger method (6, 7), gives a higher recovery of organic C and less variation in percent recovery among different groups of samples.

When external heat is applied, temperature control is extremely important. The actual temperature selected is not too critical so long as the procedure is standardized for that temperature. As temperature increases, reaction time required should decrease and precision increase.

Equations

1. Reaction of $Cr_2O_7^{2-}$ with organic matter.
   a. The $Cr_2O_7^{2-}$ will react with carbon as follows:
   $$2Cr_2O_7^{2-} + 3C^0 + 16H^+ \xrightarrow{\Delta} 4Cr^{3+} + 3CO_2 + 8H_2O$$
   b. Similarly, Cr_2O_7 will react with organic hydrogen as follows:
   $$Cr_2O_7^{2-} + 6H^0 + 8H^+ \xrightarrow{\Delta} 2Cr^{3+} + 7H_2O$$
   c. The presence of organic oxygen will decrease the amount of total carbon oxidized by the Cr_2O because of the following reaction:
   $$RCOOH \longrightarrow RH + CO_2$$
   Reaction (b) tends to compensate for the loss of C due to reaction (c) so that the assumption that each C atom is oxidized from C^0 to C^{4+} reflects the overall electron change in the reaction. Excess $Cr_2O_7^{2-}$ is back titrated with standard Fe^{2+} solution to determine the amount that has reacted.
2. Reaction of Fe^{2+} with $Cr_2O_7^{2-}$
   a. Ferrous iron reacts with $Cr_2O_7^{2-}$ as follows
   $$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \xrightarrow{\Delta} 2Cr^{3+} + 6Fe^{3+} + 7H_2$$

Three methods for determining organic matter are given below. The first is the classical Walkley-Black method (5, 6, 7). The calculation of organic matter assumes that 77% o

[1] *Wisconsin representative to NCR-13 committee.*

3. 50-mL Erlenmeyer flasks
4. Digestion oven, 90°C, with air circulation fan and fume exhaust
5. 10- and 25-mL pipettes or dispensers
6. Standard organic matter samples

Reagents

1. Digestion solution (0.5 *M* $Na_2Cr_2O_7$ in 5 *M* H_2SO_4): Dissolve 140 g $Na_2Cr_2O_7 \cdot 2\ H_2O$ in 600 mL of distilled water. Slowly add 278 mL of concentrated H_2SO_4. Allow to cool and dilute to 1 liter.

Procedure

1. Using an NCR-13 1-g scoop, scoop 1 g of soil into a 50-mL Erlenmeyer flask, using standard scooping techniques (see Chapter 2).
2. Add, by means of a pipette or dispenser, 10 mL of dichromate-sulfuric acid digestion solution. Include a reagent blank without soil.
3. Cover the Erlenmeyer flasks with glass marbles, which act as reflux condensers, to minimize loss of chromic acid.
4. Place in the digestion oven and heat to 90°C for 90 minutes.
5. Remove samples from the oven, let cool 5 to 10 minutes, remove the glass marble caps, and add 25 mL of water.
6. Mix the suspension thoroughly by blowing air through the suspension via the 25-mL pipettes used to add water or by mechanical shaking.
7. Allow to stand three hours or overnight.
8. Transfer 10 mL (or other suitable volume of clear supernatant into a colorimeter tube. This can be accomplished conveniently by use of a pipette bank set to dip a suitable distance into the supernatant solutions. Care must be taken not to disturb the sediment on the bottom of the flasks.
9. The blue color intensity of the supernatant is read on a colorimeter at 645 nm, with the reagent blank set to give 100% transmittance (or 0 absorbance). The instrument is calibrated to read percent organic matter (or tons per acre) from a standard curve prepared from soils of known organic matter content.

ALTERNATE PROCEDURE INVOLVING HEAT OF DILUTION

Reagents

1. 0.5 *M* $Na_2Cr_2O_7$: Dissolve 149 g of $Na_2Cr_2O_7 \cdot 2\ H_2O$ in water and dilute to 1 liter
2. H_2SO_4, concentrated, 96%

Procedure

1. Using an NCR-13 1-g scoop, scoop 1 g of soil into a 50-mL Erlenmeyer flask, using standard scooping techniques.
2. Add 10 mL of $Na_2Cr_2O_7$ solution by means of dispenser.
3. Add 10 mL of concentrated sulfuric acid, using a suitable dispenser. A supply of 2% $NaHCO_3$ should be readily available to neutralize spilled acid on skin, clothing, or lab bench.
4. Allow to react for 30 minutes.
5. Dilute with 15 mL of water and mix.
6. Proceed with step 7 immediately above.

A sample exchange involving 25 soil samples among 13 labs in the North Central region showed that results using the routine colorimetric procedure agreed closely with those of the Walkley-Black method. However, the standard deviation was somewhat greater with the routine colorimetric procedure, as might be expected (see Table 1). Other modifications of the Walkley-Black method gave greater amounts of variation among labs. This variation would likely have been lower had the comparisons all been made by the same lab. Nevertheless, the results underscore the need to standardize carefully whatever procedure is followed.

ORGANIC MATTER STANDARD CURVE

Analyze standard soils of known organic matter content (determined by the Walkley-Black method above or by means of a carbon analyzer) in duplicate by the routine colorimetric method above, except read absorbance on the colorimeter. Then plot the known percent organic matter (or tons organic matter, tons OM/acre) against absorbance readings. Calibrate an instrument scale in % OM (or tons OM/acre) using values obtained from the graph.

Table 1. Comparison of organic matter results determined by different modifications of the Walkley-Black method.

Comparison	Mean organic matter and std. dev.					
		Mean	SD		Mean	SD
		------%			------%	
External heat applied:						
Titration (4) vs. Colorimetric (10)	Titration	2.93	0.16	Colorimetric	2.82	0.65
Heat of dilution:						
Titration (4) vs. Colorimetric (4)	Titration	2.93	0.16	Colorimetric	2.58	0.59
Colorimetric procedures:						
Weight (4) vs. Scoop (6)	Weight	2.60	0.53	Scoop	2.97	0.70
Filter (3) vs. Settle (6)	Filter	2.15	0.26	Settle	3.21	0.54
Heat of diln. (4) vs. Ext. heat (6)	Heat of diln.	2.58	0.59	Ext. heat	2.99	0.68

Results are means of 25 samples ranging from 0.3 to 8.1% organic matter analyzed by 13 North Central region soil testing labs in 1979.

Numbers in parentheses indicate number of labs involved in each comparison.

If a rapid in-situ measurement of the apparent electrical conductivity is desired showing the extent of a saline area the non-contacting terrain conductivity meters, such as the EM31 and EM38 made by Geonics Limited[2] (4) can be used. If a more accurate reading is needed, measurement of electrical conductivity on a 1:1 soil to water suspension in the laboratory is best. Once a salt problem is identified by one of these methods the more detailed information needed to correct the situation should be obtained from electrical conductivity measurement on a saturated paste extract. Some laboratories use chloride content as an indication of salt content. This is not an acceptable method because many salt affected soils are low in chlorides but high in sulfates.

SOIL SALINITY METHODS

1. Non-contacting terrain conductivity meters

Soil electrical conductivity can be obtained from above-ground electromagnetic measurements by relating electromagnetic conductivity to electrical conductivity (1). This method is very fast and accurate once the meter is calibrated for a particular set of conditions. Several soil factors in addition to salinity, such as soil porosity, moisture and amount and type of clay influence the readings (4), therefore a single calibration can only be used on similar soils.

Refer to the instrument instruction manual for details.

2. 1:1 Soil:Water Method

The electrical conductivity value obtained by the 1:1 soil to water method is not as easily interpreted as that for the saturated paste method. With the 1:1 method the relationship between conductivity and crop growth varies with soil texture.

Equipment

1. Standard NCR-13 10-g scoop
2. Dip type conductivity cell
3. Conductivity meter

Reagents

1. Distilled water
2. Calibration solution (0.01 *M* KCl solution): Dissolve 0.7456g KCl in 1 liter of water. This solution has a conductivity of 1.41 mmhos/cm at 25°C.

Procedure

1. Using a NCR-13 10-g scoop, measure two scoops of soil into a large test tube or paper portion cup.
2. Add 20 mL of distilled water. Periodically stir the suspension and allow it to equilibrate for 15 to 20 minutes. This sample could also be used for a pH measurement after (not before) taking the conductivity measurement.
3. Insert the conductivity cell calibrated with the 0.01*M* KCl into the suspension and read the conductivity in mmhos/cm.

3. Saturated Paste Method

The saturated paste method has long been the recommended method for assessing soil salinity in relation to plant growth. The advantage of this method is that the saturation moisture percentage is directly related to the field moisture range. Conductivity by this method relates directly to plant response for all soils without adjustment for texture (7) as with the 1:1 method. The disadvantage of this method is more expense and time.

Equipment

1. Conductivity meter
2. Conductivity cell
3. 250-mL containers (such as plastic cups)
4. Buchner funnels

Reagents

1. Distilled water
2. 0.01 *M* KCl solution: Dissolve 0.7456g KCl in 1 liter of water. This solution has a conductivity of 1.41 mmhos/cm at 25°C.

Procedure

1. The amount of soil used will depend on the number of measurements that will be made on the extract. A 250-g sample provides sufficient extract for most purposes.
2. Add distilled water to the soil while stirring it with a spatula. At saturation the soil paste will glisten as it reflects light, flow slightly when the container is tipped, and the paste slides freely and cleanly off the spatula for all soils except clays.
3. After mixing allow the sample to stand for at least 1 hour and then recheck for saturation. Free water

[2]*Use of product name does not constitute endorsement.*

Table 1. The relationship between conductivity and degree of salinity for the 1:1 method and the saturated paste method.

	Degree of Salinity				
Texture	Non-Saline	Slightly Saline	Moderately Saline	Strongly Saline	Very Strongly Saline
1:1 Method	mmhos/cm				
Coarse sand to loamy sand	0-1.1	1.2-2.4	2.5-4.4	4.5- 8.9	9.0 +
Loamy fine sand to loam	0-1.2	1.3-2.4	2.5-4.7	4.8- 9.4	9.5 +
Silt loam to clay loam	0-1.3	1.4-2.5	2.6-5.0	5.1-10.0	10.1 +
Silty clay loam to clay	0-1.4	1.5-2.8	2.9-5.7	5.8-11.4	11.5 +
Saturated Paste Method	mmhos/cm				
All textures	0-2.0	2.1-4.0	4.1-8.0	8.1-16.0	16.1 +

and analyzed quickly. The water-holding characteristics of the various growth media tend to be related to the bulk density. This acts as an automatic compensator for differences in bulk densities which affect interpretation of results from saturation extracts. As demonstrated by Geraldson (3), nutrient balance is very important in weakly buffered systems such as exist with many greenhouse growth media. With the saturation extract approach, nutrient balance information is readily calculated. Growth media which contain slow-release fertilizer can be extracted by the saturation extract method with very little inflation of the test results (13). With other handling and extraction procedures test values are greatly inflated due to excessive solubilization of the slow-release fertilizer.

Available micronutrient levels in plant growth media are important for the growth of container grown plants. In peat and bark based growth media, the base micronutrients are complexed by organic compounds (10). Hence, the concentrations of these micronutrients in the water saturation extract are quite low. Zinc and manganese concentrations rarely exceed 0.8 mg/L and iron rarely exceeds 4.0 mg/L. Therefore, it is difficult to distinguish between deficient and adequate levels.

In evaluating fifteen extractants Berghage et al. (1) found that extractable levels of iron, maganese and zinc could be increased greatly by using weak solutions of various salts, acids or chelates in the saturating solution with the saturation extract procedure. A 0.005 *M* DTPA was found to most consistently increase extractable micronutrient levels while having only a minor effect on the other key test parameters: total soluble salts and extractable levels of nitrate, phosphorus, potassium, calcium, magnesium sodium and chloride.

SATURATED MEDIA EXTRACT (SME) METHOD

This procedure was developed at Michigan State University and has been routinely used in their soil testing lab. It allows extraction of moist samples just as they come from greenhouses. Drying of samples is unnecessary and undesirable. Storage of greenhouse growth media in either the dry or moist state will influence the soluble nitrate-nitrogen and soluble salt levels. If samples will not be extracted within two hours of receipt, store them in a refrigerated area.

Equipment

1. 600-mL plastic beaker
2. Spatula
3. Buchner funnel, 11-cm
4. Filter paper (Whatman No. 1), 11-cm
5. Vacuum flask, 500-mL
6. Vacuum pump
7. Vial, snap-cap 100-mL
8. Conductivity meter, (Solu-bridge 31 or equivalent)
9. Conductivity cell, dipping type, cell constant = 1.0
10. Thermometer
11. pH meter with expanded scale or specific ion meter
12. pH glass electrode with a paired calomel reference electrode
13. Nitrate electrode with paired reference electrode
14. Colorimeter
15. Flame emission, atomic absorption, and/or plasma emission spectrophotometer
16. Volumetric flasks and pipettes as required for preparation of reagents and standard solutions.

Reagents

1. Distilled water
2. 0.01 *M* potassium chloride (for standardizing solu-bridge)
3. Reagents for determining pH, nitrate-N, phosphorus, potassium, calcium and magnesium.

Procedure

1. Fill a 600-mL beaker about 2/3 full with the growth medium. Gradually add distilled water while mixing until the sample is just saturated. At saturation the sample will flow slightly when the container is tipped and is easy to work with a spatula. After mixing, allow the sample to equilibrate for one hour and then recheck the criteria for saturation. The saturation sample should have no appreciable free water on the surface nor should it have stiffened. Adjust as necessary by addition of growth medium or distilled water. Then allow to equilibrate for an additional half hour.
2. Determine the pH of the saturated sample by carefully inserting the electrodes. Wiggle the electrodes gently to attain good solution contact.
3. Attach a Buchner funnel lined with filter paper to a vacuum flask. Apply a vacuum and transfer the saturated sample into the Buchner funnel. Work sample with a spatula and tap the funnel to eliminate entrapped air and insure good contact between the saturated sample and the filter. Continue vacuum, collecting the extract in the flask. No more than 15 minutes of vacuum should be required. Transfer the extract to the snap-cap vial. All subsequent analyses are done on the extracted solution.
4. Check the temperature of the extract and adjust the temperature dial on the solu-bridge. Rinse the electrode and dip the conductivity cell into the extract. Determine the electrical conductivity of the extract and record in mS per cm. Use 0.01 *M* KCl to calibrate the solu-bridge. Prepare the 0.01 *M* KCl solution by dissolving exactly 0.7456 g KCl in 800 ml of distilled-deionzed water and diluting it to 1 liter. With the temperature adjustment properly made, a 0.01 *M* KCl solution should give a solu-bridge reading of 1.418 mS per cm.
5. After establishing the standard curve, determine the nitrate-nitrogen content with a nitrate electrode. Record millivolt reading on an expanded scale pH meter or specific ion meter, and obtain the concentration of nitrate from a standard curve of Emf vs. nitrate concentration plotted on semilogarithmic graph paper. (See Chapter 5).
6. Determine phosphorus on a aliquot of the extract by one of the accepted colorimetric procedures. (See Chapter 6). Determine potassium, calcium and magnesium on an aliquot of the extract by flame emission or atomic absorption spectroscopy (See Chapter7).

Modified (DTPA) Saturated Media Extract Method

By using 0.005 *M* DTPA as the primary saturating solution, extraction of the basic micronutrients (zinc. manganese and iron) can be greatly enhanced. For each liter of DTPA solution to be prepared transfer exactly 1.97 g dry DTPA (diethylenetriaminepenta-acetic acid) into a 1 liter volumetric flask and add about 800 mL of pure water. Heating the water to 50°C and stirring facilitates dissolution of the DTPA. After the solution has cooled make to volume with pure water. The modified SME method involves a change in the procedure used to saturate the growth media and in the measurement of pH.

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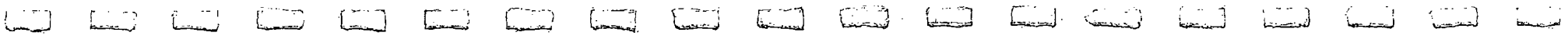

DETERMINATION OF EXCHANGEABLE SODIUM

A. Reagents

1. Extracting Solution (1N NH_4OAc): Use the same extracting solution used for the Cu and Fe test (plastic bottles).

2. Standard Sodium Solution: Dissolve 2.541 grams sodium chloride (NaCl) in extracting solution and dilute to 1 liter. This solution contains 1000 ppm Na and serves as the stock solution. Dilute 100 ml to 1 liter with extracting solution. This solution contains 100 ppm Na. Other sodium standards are prepared as follows:

   To each of four 100 volumetric flasks, add 0, 1.0, 2.0, 3.0, and 4.0 ml of the 100 ppm stock solution. Bring to volume with extracting solution. This will give standards of 0, 1, 2, 3, and 4 ppm Na.

B. Determination

Scoop 1 g of soil into a plastic shaking bottle, add 10 ml of extracting solution from an automatic dispensing pipette and shake at 250 OPM in an oscillating shaker for 5 minutes. Pour into centrifuge tubes and spin 15 minutes. Care must be taken to obtain a clear filtrate. Determine the #/A in each filtrate by atomic emission. If the reading exceeds 80 #/A, dilute the sample and redetermine the concentration.

C. AA Standardization

Auto-zero on the "0 ppm" standard. Set the AA spectrophotometer so that $S_1 = 20$, $S_2 = 40$, and $S_3 = 60$ #/A. Aspirate the 1 ppm solution and standardize S_1; aspirate the 2 ppm solution and standardize S_2; aspirate the 3 ppm solution and standardize S_3. Check the standardization.

DETERMINATION OF SOLUBLE SALTS

A. Reagents

1. 0.01N Potassium Chloride Solution
Dissolve 0.745 grams potassium chloride (KCl) in 1 liter distilled water. This solution is used for checking the accuracy of the conductivity instrument and cell. With the temperature compensator properly set, the instrument should read 141 mhos x 10^{-5}.

B. Determination

Measure 5 grams of soil into a 1 ounce paper cup, add 10 ml of distilled water, stir, and let stand for 60 minutes. Filter. Suction some of the liquid into the Solu-Bridge electrode and read the K-value. When testing organic soils, use 5 grams of soil and 20 ml of water, multiply the K-readings by 2 to get the actual K-values.

C. Interpretation of Soil Conductivity Reading

Listed below are a series of values of K (specific conductance in mhos x 10^{-5} at 25° C) typical of different soil and plant conditions. These data were obtained by measuring 1:2 soil extracts; that is, 1 part of soil to 2 parts by weight of water.

SOIL AND PLANT CONDITIONS**	K VALUES	
Unfertilized, leached, field soils	below 15	
Well fertilized soils for optimum plant growth	100-200	
Soluble salt content critical for growth of salt sensitive crops	above 200	Applicable to Greenhouse soils
Severe injury to plants	above 300	
Soluble salt content critical for germination	above 100	See note below
Critical salt content of soils flooded with sea water	100	See note below

NOTE: The toxicity of a single salt like sodium chloride is greater than that of an equivalent amount of a mixture of salts. For this reason, the injury of salts to sensitive plants growing in soils flooded with sea water begins at K value of about 100. To obtain the amount of salt (sodium chloride) in lbs per acre-six-inches (2,000,000 lbs of soil) in flooded soils, multiply K by 20. For example, K of 100 is equivalent to 2000 lbs of salt (sodium chloride) per acre-six-inches).

** These figures were obtained from unpublished data of the New York State Agricultural Experiment Station, Department of Agronomy.

for Heavy Metals

NITRIC-PERCHLORIC ACID DIGESTION FOR ICP

Weigh 0.300 gram sample of sludge, plant, soil, etc. into a 75 ml digestion tube. Add 3 ml of 1:1 HNO_3-$HClO_4$ mixture. Let set overnight, then set the tube rack in the cold heating block and start digestion cycle. Place a small funnel in the top of the tube to act as a condenser. Heat on the Tecator block programmed as follows: 2:20 hr - 130° C; 3:40 hr - 185° C; 4:00 hr - 210 °C, or heat for 2 hours at 130 °C on the Technicon block, then increase temperature to 205° C and heat for 2 more hours. Digest should be colorless, or very pale yellow. Transfer digest with DI water to either a 10 ml or 25 ml volumetric flask. Dilute to volume and analyze on the ICP.

Usually the clear digest may be transferred to the small centrifuge tube without agitation. If silica is transferred, the diluted digest should be centrifuged for 5 minutes at 2700 RPM.

Final Action

7.021 *Principle*

Automation of macro Kjeldahl method is in 6 steps: sample and reagent addn, initial and final digestion, cooling and diln, NaOH addn, steam distn and titrn, and automatic pumping of flask contents to waste. Chemistry is carried out in macro Kjeldahl flasks equipped with side arms which are rotated at 3 min intervals thru each successive step.

7.022 *Apparatus*

(a) *Kjeldahl (protein/nitrogen) analyzer.*—Kjel-Foss Automatic, Model 16210 (Foss America, Inc., PO Box 504, Rt 82, Fishkill, NY 12524), or equiv.

(b) *Weighing papers.*—120 × 120 mm N-free tissues, Foss America, Inc., or equiv.

7.023 *Reagents*

(a) *Kjel-tabs.*—Contg 5 g K_2SO_4 and 0.25 g HgO (Foss America, Inc.).

(b) *Kjeldahl (protein/nitrogen) analyzer reagents.*—Prep. following according to manufacturer's instructions: (*1*) *Sulfuric acid.*—96–98%. (*2*) *Hydrogen peroxide.*—30–35%. (*3*) *Ammonium sulfate std solns.*—(*a*) *Std soln I.*—Dissolve 30.000 ± 0.030 g $(NH_4)_2SO_4$ in H_2O and dil. to 1 L with H_2O. (*b*) *Std soln II.*—Dissolve 0.750 ± 0.001 g $(NH_4)_2SO_4$ in H_2O and dil. to 1 L with H_2O. (*4*) *Mixed indicator soln.*—Dissolve 1.000 g Me red and 0.250 g methylene blue in alcohol and dil. to 1 L with alcohol. Dil. 10 mL this soln to 1 L with H_2O. (*5*) *Sodium hydroxide-sodium thiosulfate soln.*—40% NaOH-8% $Na_2S_2O_3 \cdot 5H_2O$. (*6*) *Dilute sulfuric acid soln.*—0.6%. Dil. 30 mL 96–98% H_2SO_4 to 5 L with H_2O.

7.024 *Determination*

(*Caution: See* **51.019, 51.030, 51.065,** and **51.070.**)

Place 3 Kjel-tabs in special flask (500 mL of design compatible to Foss instrument) in position 1. Shift dispenser arm over flask and depress H_2SO_4 lever, initiating simultaneous addn of 10 mL 30–35% H_2O_2 and 12–15 mL 96–98% H_2SO_4 (depending on fat content of sample). To flask, add accurately weighed sample (ca 1.0 g if <45% protein, and ca 0.5 g if >45% protein) wrapped in weighing paper and close lid. Flask automatically rotates to position 2 where sample digests 3 min, and then to position 3 for 3 min addnl digestion. In position 4, flask is cooled by centrifugal blower, lid opens automatically, and 140 mL H_2O is added automatically. Flask rotates to position 5, where NaOH-$Na_2S_2O_3$ soln is automatically introduced in excess. Released NH_3 is steam distd quant. into 200 mL tall-form titrn beaker contg 50 mL mixed indicator soln, and is simultaneously titrd automatically with dil. H_2SO_4 soln delivered by photometrically regulated syringe. Final position of syringe is measured by potentiometer, output of which feeds electronic circuitry for conversion to visual display and/or printout in % N or % protein with appropriate conversion factors. In position 6, flask is emptied. Calibrate instrument initially each day with aliquots of $(NH_4)_2SO_4$ std solns and check periodically as stated in operating manual.

Ref.: JAOAC **59**, 141(1976).

For minerals / plant tissue

STANDARD DRY ASH ANALYSIS FOR ICP

Weigh a 1.0 g sample of feed or plant sample into a high-form silica crucible. Dry ash at 500° C for a minimum of 4 hours. Transfer ash to 50 ml centrifuge tube, washing and rinsing the crucible with 2.4N HNO_3. Dilute the sample to the 25.0 ml mark on the tube with 2.4N HNO_3. Shake on a reciprocating or vortex shaker for 10 minutes. Let ash settle. Transfer an aliquot to a small centrifuge tube. Centrifuge for 10 minutes at 2700 RPM. Analyze the supernatent solution on the calibrated ICP, taking care not to lower the probe into the ash.

APPENDIX B

SOIL AND PLANT ANALYSES DATA

LAB NUMBER 25802
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01685

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 1

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST	NO CROP GIVEN						
1990	NO CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.
AND ONLY THE FOLLOWING GENERAL LIME RECOMENDATION CAN BE GIVEN-
FOR A DESIRED PH OF 6.5, ADD 3.0 TONS OF LIME PER ACRE,
FOR A DESIRED PH OF 6.0, ADD 2.5 TONS OF LIME PER ACRE.

Exchangeable sodium = 28 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3 3

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3-N lb/A	ORGANIC MATTER %	SOLUBLE SALTS Mhos X10³
8	.0	5.1	66	116	224	3170	263	14	56	8	2.0	189	.	.	.	4.8		.	71

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 22 MG/KG NICKEL 16 MG/KG COPPER 6 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 21 MG/KG ZINC 73 MG/KG

GROWER COPY

LAB NUMBER 25803
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01686

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 2

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO	CROP GIVEN						
1990 NO	CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE,
AND ONLY THE FOLLOWING GENERAL LIME RECOMENDATION CAN BE GIVEN-
FOR A DESIRED PH OF 6.5, ADD 1.0 TONS OF LIME PER ACRE,
FOR A DESIRED PH OF 6.0, ADD 1.0 TONS OF LIME PER ACRE.

Exchangeable sodium = 96 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3. .3

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3 - N lb/A	ORGANIC MATTER %	SOLUB SALTS Mhos X10
8	.0	5.7	68	96	234	3850	253	13	72	8	2.2	165	.	.	.	35.0		.	42

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 18 MG/KG NICKEL 11 MG/KG COPPER 6 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 20 MG/KG ZINC 51 MG/KG

GROWER COPY

LAB NUMBER 25804
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01687

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 3A

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO	CROP GIVEN						
1990 NO	CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.

NO LIME IS NEEDED NOW.

Exchangeable sodium = 195 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3. 3

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3-N lb/A	ORGANIC MATTER %	SOLUBL SALTS Mhos X10
0	.0	7.7	70	18	49	2550	176	15	89	10	.9	29				62.7			20

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD < 13 MG/KG NICKEL 12 MG/KG COPPER 3 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 14 MG/KG ZINC 41 MG/KG

GROWER COPY

LAB NUMBER 25805
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01688

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 3B

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST	NO CROP GIVEN						
1990	NO CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.

NO LIME IS NEEDED NOW.

Exchangeable sodium = 440 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3. .

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca. lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3-N lb/A	ORGANIC MATTER %	SOLU SA MI XI
0	.0	7.8	70	<2	174	5010	415	15	87	12	1.5	23	.	.	.	81.9		.	

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 36 MG/KG NICKEL 51 MG/KG COPPER 25 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 17 MG/KG ZINC 105 MG/KG

GROWER COPY

LAB NUMBER 25806
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01689

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 3C

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO	CROP GIVEN						
1990 NO	CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.

NO LIME IS NEEDED NOW.

Exchangeable sodium = 288 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3, ,3

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	BASE SATURATION % Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3-N lb/A	ORGANIC MATTER %	SOLUBLE SALT Mhos X10
0	.0	6.8	70	76	164	4680	307	13	89	10	1.6	69	.	.	.	85.8		.	3

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 19 MG/KG NICKEL 13 MG/KG COPPER 7 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 18 MG/KG ZINC 59 MG/KG

GROWER COPY

LAB NUMBER 25807
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01690

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 4

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO	CROP GIVEN						
1990 NO	CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE,
AND ONLY THE FOLLOWING GENERAL LIME RECOMENDATION CAN BE GIVEN-
FOR A DESIRED PH OF 6.5, ADD 1.0 TONS OF LIME PER ACRE,
FOR A DESIRED PH OF 6.0, ADD .0 TONS OF LIME PER ACRE.

Exchangeable sodium = 48 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3. .3

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3 - N lb/A	ORGANIC MATTER %	SOLUB SALT Mho X10
8	.0	6.0	68	114	302	4440	319	15	73	9	2.5	113	.	.	.	14.7		.	3

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 23 MG/KG NICKEL 14 MG/KG COPPER 9 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 20 MG/KG ZINC 66 MG/KG

GROWER COPY

LAB NUMBER 25808
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01691

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 5

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P205 LB/A	POTASH K20 LB/A	COMMENTS SEE BELOW
LAST	NO CROP GIVEN						
1990	NO CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.

NO LIME IS NEEDED NOW.

Exchangeable sodium = 200 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3. .3

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	BASE SATURATION % Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3 - N lb/A	ORGANIC MATTER %	SOLUBLE SALT Mho X10
0	.0	6.3	70	112	314	3030	241	9	84	11	4.5	106	.	.	.	91.8		.	7

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 17 MG/KG NICKEL 17 MG/KG COPPER 6 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 24 MG/KG ZINC 53 MG/KG

GROWER COPY

LAB NUMBER 25809
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01692

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development-Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 6

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO	CROP GIVEN						
1990 NO	CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE,
AND ONLY THE FOLLOWING GENERAL LIME RECOMENDATION CAN BE GIVEN-
FOR A DESIRED PH OF 7.0, ADD 4.5 TONS OF LIME PER ACRE.
FOR A DESIRED PH OF 6.5, ADD 4.0 TONS OF LIME PER ACRE.
FOR A DESIRED PH OF 6.0, ADD 3.0 TONS OF LIME PER ACRE.

Exchangeable sodium = 21 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3, .1

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3 - N lb/A	ORGANIC MATTER %	SOLUB SALT Mhos X10
6	.0	5.5	65	104	274	4630	392	20	59	8	1.8	47	.	.	.	3.8		.	2

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 22 MG/KG NICKEL 14 MG/KG COPPER 12 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 19 MG/KG ZINC 74 MG/KG

GROWER COPY

LAB NUMBER 25810
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01693

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 7

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO CROP GIVEN							
1990	NO CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.
AND ONLY THE FOLLOWING GENERAL LIME RECOMENDATION CAN BE GIVEN-
FOR A DESIRED PH OF 7.0, ADD 3.5 TONS OF LIME PER ACRE.
FOR A DESIRED PH OF 6.5, ADD 3.0 TONS OF LIME PER ACRE.
FOR A DESIRED PH OF 6.0, ADD 2.5 TONS OF LIME PER ACRE.

Exchangeable sodium = 23 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3 , 1

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	BASE SATURATION % Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3 - N lb/A	ORGANIC MATTER %	SOLUB SALTS Mhos X10
8	.0	5.2	66	68	155	3310	271	14	57	8	1.4	85	.	.	.	2.0		.	3

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 21 MG/KG NICKEL 14 MG/KG COPPER 7 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 18 MG/KG ZINC 64 MG/KG

GROWER COPY

LAB NUMBER 25811
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01694

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 8

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO CROP GIVEN							
1990 NO CROP GIVEN							Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.
AND ONLY THE FOLLOWING GENERAL LIME RECOMENDATION CAN BE GIVEN-
FOR A DESIRED PH OF 6.5, ADD 3.0 TONS OF LIME PER ACRE,
FOR A DESIRED PH OF 6.0, ADD 2.5 TONS OF LIME PER ACRE.

Exchangeable sodium = 22 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIM RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3 .2

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca. lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3 - N lb/A	ORGANIC MATTER %	SOLUBLE SALTS Mhos X10
8	.0	5.6	66	172	230	3630	345	16	58	9	1.9	27	.	.	.	2.5		.	2

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 23 MG/KG NICKEL 16 MG/KG COPPER 12 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 19 MG/KG ZINC 59 MG/KG

GROWER COPY

LAB NUMBER 25812
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01695

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 9

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO	CROP GIVEN						
1990 NO	CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE.

NO LIME IS NEEDED NOW.

Exchangeable sodium = 65 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3, ,2

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3-N lb/A	ORGANIC MATTER %	SOLUBLE SALT Mho X10
0	.0	7.3	70	118	180	6250	466	18	88	11	1.3	23	.	.	.	19.6		.	3

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD	24 MG/KG	NICKEL	18 MG/KG	COPPER	20 MG/KG
CADMIUM	<.33 MG/KG	CHROMIUM	18 MG/KG	ZINC	64 MG/KG

GROWER COPY

LAB NUMBER 25813
REFER TO LAB NUMBER TO IDENTIFY
SAMPLE IN FUTURE CORRESPONDENCE.
SOIL BAG NUMBER IL01696

The Ohio State University
Research-Extension Analytical Lab
The Ohio Agricultural Research and Development Center
Wooster, Ohio 44691

LIME AND FERTILIZER RECOMMENDATIONS

YOUR SAMPLE ID 10

ANNUAL RECOMMENDATION

ACRES REPRESENTED 0

YEAR	CROP	YIELD GOAL	LIME T/A	NITROGEN N LB/A	PHOSPHATE P2O5 LB/A	POTASH K2O LB/A	COMMENTS SEE BELOW
LAST NO	CROP GIVEN						
1990 NO	CROP GIVEN						Y

Y SINCE THE CROP FOR THIS YEAR WAS NOT GIVEN, A FERTILIZER RECOMMENDATION CAN NOT BE MADE,
AND ONLY THE FOLLOWING GENERAL LIME RECOMENDATION CAN BE GIVEN-
FOR A DESIRED PH OF 6.5, ADD 2.0 TONS OF LIME PER ACRE,
FOR A DESIRED PH OF 6.0, ADD 1.5 TONS OF LIME PER ACRE.

Exchangeable sodium = 43 #/A

BATTELLE-T ZWICK
505 KING AVE
COLUMBUS OHIO 43201

COUNTY FRANKLIN RECEIVED SAMPLE 9/21/89 DATE PRINTED TUE, OCT 3, 1989 PLAN 3. 2

SAMPLE INFORMATION		STANDARD TEST RESULTS										SPECIAL TESTS RESULTS							
									BASE SATURATION										
PLOW DEPTH INCHES	LIME APPLIED IN LAST 2 YRS. T/A	pH	LIME TEST INDEX	PHOSPHORUS P lb/A	POTASSIUM K lb/A	CALCIUM Ca lb/A	MAGNESIUM Mg lb/A	CATION EXCHANGE CAPACITY meq/100g	% Ca	% Mg	% K	MANGANESE Mn lb/A	IRON Fe lb/A	ZINC Zn lb/A	COPPER Cu lb/A	BORON B lb/A	NITRATES NO_3-N lb/A	ORGANIC MATTER %	SOLUBLE SALT Mho X10
8	.0	5.8	67	116	308	6180	522	22	71	10	1.8	59	.	.	.	5.6		.	3

THIS FINAL REPORT INCLUDES RESULTS FOR ALL STANDARD AND SPECIAL TESTS

LEAD 29 MG/KG NICKEL 21 MG/KG COPPER 14 MG/KG
CADMIUM <.33 MG/KG CHROMIUM 21 MG/KG ZINC 80 MG/KG

GROWER COPY

T. ZWICK/BATTELLE

(RESULTS ARE REPORTED IN MICROGRAM / GRAM OF SOLID)

NOTE: < INDICATES THAT THE RESULT IS LESS THAN THE GIVEN VALUE

DATE OF ANALYSIS: 9/26/89

	SAMPLE	% N	P	K	CA	MG	MN	FE	B	CU
1	CHECK		4772	20012	12216	4993	0.772	122.6	0.862	0.51
2	1	2.80	2751	21335	5176	1886	81.62	75.45	94.60	6.95
3	2	2.82	3187	29038	5123	1447	94.88	98.63	876.2	8.49
4	3	2.47	2113	32870	4842	1368	52.47	137.6	1735	4.98
5	4	2.80	2885	27551	4954	1359	75.98	82.65	284.6	8.29
6	5	2.58	2366	30744	4244	1531	66.18	150.4	1668	6.26
7	6	2.53	2843	20835	6245	2572	58.39	80.98	30.44	9.15
8	7	2.48	2852	19986	5924	2349	97.55	76.58	23.62	7.49
9	8	2.87	3222	17909	7144	3207	59.82	86.63	21.03	9.32
10	CHECK		4844	19892	12153	5004	0.769	123.4	4.688	< 0.50
11	9	3.12	3247	19329	6223	2575	28.32	94.37	166.2	10.5
12	10	3.08	2999	15403	6627	2798	45.69	100.6	18.64	8.78
13	CK-B		2591	19808	5161	2748	70.20	106.2	9.144	8.40

	SAMPLE	ZN	AL	NA	Pb	Cd	Ni	Cr
1	CHECK	0.612	4.250	3.938				
2	1	32.39	20.12	16.40	6.4	< 0.2	2.1	3.7
3	2	28.56	27.39	23.80	< 6.0	< 0.2	3.1	2.9
4	3	14.16	53.29	40.54	7.6	< 0.2	< 2.0	2.1
5	4	32.43	14.08	16.59	< 6.0	< 0.2	3.9	3.1
6	5	18.23	68.46	32.58	< 6.0	< 0.2	< 2.0	1.8
7	6	29.92	23.84	16.59	< 6.0	< 0.2	3.1	1.9
8	7	36.43	18.13	19.37	< 6.0	< 0.2	5.0	1.6
9	8	49.79	21.12	14.98	< 6.0	< 0.2	< 2.0	3.5
10	CHECK	0.453	5.977	5.290				
11	9	34.28	20.49	12.16	< 6.0	< 0.2	< 2.0	2.9
12	10	49.96	16.40	15.35	< 6.0	< 0.2	< 2.0	1.4
13	CK-B	33.12	51.43	11.03				

READY